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Ta–Nb ORDER IN THE CRYSTAL STRUCTURE OF NIOBIUM-RICH CALCIOTANTITE

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Abstract

The crystal structure of niobium-rich calciotantite, Ca ($Ta_{2.82}$ $Nb_{1.18}$) O_{11}, hexagonal, *a* 6.2261(6), *c* 12.280(1) Å, *V* 412.18(8) Å^3, space group $P6_322$, $Z = 1$, $D_{calc.} = 6.735$ g/cm^3, has been refined to an *R* index of 1.8% based on 414 observed (4σ) reflections measured with Mo*K*α X-radiation. There is one unique *Ca* site occupied by Ca and coordinated by eight O-atoms with a <*Ca*–O> distance of 2.512 Å. There are two unique *Ta* sites both occupied by Ta and Nb; the *Ta*(1) site is coordinated by seven O atoms in a distorted pentagonal dipyramidal arrangement, and the *Ta*(2) site is coordinated by six O atoms in a distorted octahedral arrangement. The *Ta*(2)O_6 and *Ca*O_8 polyhedra occupy alternate vertices of a 6^3 net to form an open network of edge-sharing polyhedra of the form [$CaTaO_8$]. The *Ta*(1)O_7 polyhedra share four of their pentagonal edges to form a [Ta_3O_{11}] sheet with triangular holes at the vertices of a 6^3 net. These sheets alternate along [001] to form a dense framework structure. The structure of calciotantite is topologically similar to that of natrotantite, $Na_2Ta_4O_{11}$; the [Ta_3O_{11}] sheets are topologically identical in both structures, and the [$CaTaO_8$] and [Na_2TaO_8] sheets are the same if they are written as [□$CaTaO_8$] and [Na_2TaO_8]. The [Ta_3O_{11}] sheet is also topologically identical to the [$(UO_2)_3\phi_5$] (ϕ = O,OH) in billietite, protasite, becquerelite and α–U_3O_8. Niobium is slightly preferentially ordered (relative to Ta) at the *Ta*(1) site, indicating a slight difference in the crystal-chemical behavior of Ta and Nb.

Keywords: calciotantite, crystal structure, Ta–Nb order, natrotantite.

Sommaire

Nous avons affiné la structure cristalline d'un échantillon de calciotantite niobifère, Ca ($Ta_{2.82}$ $Nb_{1.18}$) O_{11}, hexagonale, *a* 6.2261(6), *c* 12.280(1) Å, *V* 412.18(8) Å^3, groupe spatial $P6_322$, $Z = 1$, $D_{calc.} = 6.735$ g/cm^3, jusqu'à un résidu *R* de 1.8% en utilisant 414 réflexions observées (4σ), mesurées avec rayonnement Mo*K*α. La structure contient un site unique *Ca*, qu'occupe le Ca, en coordinence avec huit atomes d'oxygène ayant une distance <*Ca*–O> de 2.512 Å. Elle contient deux sites distincts *Ta*, sur lesquels sont répartis à la fois Ta et Nb. Le site *Ta*(1) a une coordinence sept, avec des atomes d'oxygène agencés en bipyramide pentagonale difforme. En revanche, le site *Ta*(2) est coordonné à six atomes d'oxygène définissant une agencement octaédrique difforme. Les polyèdres *Ta*(2)O_6 et *Ca*O_8 sont disposés en alternance aux nodes d'un réseau 6^3 pour former une trame ouverte de polyèdres à arêtes partagées ayant une stoechiométrie [$CaTaO_8$]. Les polyèdres *Ta*(1)O_7 partagent quatre des arêtes des pentagones pour former un feuillet [Ta_3O_{11}] ayant des trous triangulaires aux nodes d'un réseau 6^3. Ces feuillets alternent le long de [001] et forment une trame assez dense. La structure de la calciotantite ressemble topologiquement à celle de la natrotantite, $Na_2Ta_4O_{11}$; les feuillets [Ta_3O_{11}] sont topologiquement identiques dans les deux structures, et les feuillets [$CaTaO_8$] et [Na_2TaO_8] sont les mêmes s'ils sont exprimés sous forme [□$CaTaO_8$] et [Na_2TaO_8]. Le feuillet [Ta_3O_{11}] est aussi topologiquement identique au feuillet [$(UO_2)_3\phi_5$] (ϕ = O,OH) de la billietite, protasite, becquerelite et α–U_3O_8. Le niobium est légèrement ordonné sur le site *Ta*(1) par rapport au tantale, ce qui confirme une légère différence dans le comportement cristallochimique du Ta et du Nb.

(Traduit par la Rédaction)

Mots-clés: calciotantite, structure cristalline, mise en ordre Ta–Nb, natrotantite.

Introduction

Calciotantite was described as a new mineral by Voloshin *et al.* (1982). It is hexagonal, and the type material has the composition ($Ca_{0.99}$ $Na_{0.01}$) ($Ta_{3.20}$ $Nb_{0.80}$) O_{11}, ideally $CaTa_4O_{11}$. Voloshin *et al.* (1985) described a Na- and Pb-bearing phase, "ungursaite", from eastern Kazakhstan, but this was later shown to be calciotantite (Yamnova *et al.* 1988). The crystal structure of synthetic $CaTa_4O_{11}$ was solved by Isobe *et al.* (1975), and the cell dimensions and space group indicate that it is isostructural with calciotantite. Smeds *et al.* (1999) reported the presence of calciotantite from the northern Nyköpingsgruvan granitic pegmatite on the

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island of Utö, Stockholm Archipelago, Sweden, significantly enriched in Nb relative to the original material of Voloshin *et al.* (1982). The $CaTa_4O_{11}$ structure has two *Ta* sites, one [7]-coordinated and the other [6]-coordinated. The discovery of a Nb-enriched calciotantite provides an ideal opportunity to look for any discernable difference between the crystal-chemical behavior of Ta and Nb in an oxide environment.

Experimental

A small crystal of Nyköpingsgruvan calciotantite was extracted from a polished section, attached to a glass fiber and mounted on a Siemens *P*4 automated four-circle diffractometer equipped with Mo$K\alpha$ X-radiation. Forty-eight reflections over the range $7 \le 2\theta \le 28°$ were centered, and the unit-cell dimensions (Table 1) were refined by least-squares from the resultant setting angles. Intensity data were collected in θ:2θ scan-mode at a fixed scan-rate of 2.0°2θ/min. A total of 2608 reflections was measured over the range $4 \le 2\theta \le 60°$, with index ranges $0 \le h \le 8$, $\bar{8} \le k \le 8$, $\bar{17} \le l \le 17$. Two standard reflections were monitored every fifty-eight reflections; there were no significant changes in their intensities during data collection. The data were corrected for absorption (psi-scan method), Lorentz, polarization and background effects, averaged and reduced to structure factors; 414 reflections were considered as observed [$|F_o| \ge 4\sigma F_o$].

TABLE 1. MISCELLANEOUS INFORMATION FOR NIOBIUM-RICH CALCIOTANTITE FROM NYKÖPINGSGRUVAN

a (Å)	6.2261(6)	crystal size (mm)	0.04 x 0.06 x 0.06
c	12.280(1)	Radiation	Mo$K\alpha$/Graphite
V (Å^3)	412.18(8)	Total no. of I	2608
Sp. Gr.	$P6_322$	No. of F_o^2	414
Z	2	R(merge) %	4.2
μ (mm^{-1})	39.6	wR_2 (F_o^2) %	3.8
D_{calc} (g.cm^{-3})	6.735	R_1 ($\|F_o\| > 4\sigma$) %	1.8

Cell contents: 2 [Ca ($Ta_{2.82}$ $Nb_{1.18}$) O_{11}]

$R_1 = \Sigma(||F_o|-|F_c||)/\Sigma|F_o|$

$wR_2 = [\Sigma w(F_o^2 - F_c^2)^2 / \Sigma w(F_o^2)^2]^{1/2}$, $w = 1 / [\sigma^2(F_o^2) + (a \times P)^2 + (b \times P)]$ where a = 0.0149, b = 0.85, $P = [\{\max(0, F_o^2)\} + 2F_c^2] / 3$

An extinction correction X was refined according to the equation $F_c^* = kF_c [1 + 0.001 \times X \times F_c^2 \times \lambda^3/\sin(2\theta)]^{-1/4}$, where k is the scale factor, X = 0.0008 and k = 0.05367

Structure Solution and Refinement

Scattering curves for neutral atoms were taken from the International Tables for Crystallography (1992). *R* indices are of the form given in Table 1, and are expressed as percentages. The Siemens SHELXTL PLUS (PC version) system of programs was used for this work.

The structure refined rapidly to an *R* index of ~2% for a model with variable scattering at the *Ta*(1) and *Ta*(2) sites and anisotropic displacements on all atoms. However, the refined scattering at the *Ta*(1) and *Ta*(2) sites indicated a bulk composition (~$Ta_{2.3}Nb_{1.7}$) significantly different from that determined on the crystal by electron-microprobe analysis prior to extraction of the crystal from the polished section. The crystal was analyzed after collection of the X-ray intensity data, but the composition was not significantly different from that indicated by the initial electron-microprobe analysis. We experimented with different absorption corrections (including no absorption correction), but the resultant changes in bulk composition indicated by the site-scattering refinement were small compared with the discrepancy between the compositions indicated by the results of electron-microprobe analysis and site-scattering refinement. To deal with this problem, we constrained the refined site-populations at *Ta*(1) and *Ta*(2) to sum to the composition determined by electron-microprobe analysis. Refinement converged to an *R* index of 1.8% (as compared to 1.7% for the unconstrained refinement), and the relative ordering of Ta and Nb over *Ta*(1) and *Ta*(2) was the same for both types of refinement. Positional and displacement parameters for the constrained refinement are given in Table 2, selected interatomic distances in Table 3, and refined site-scattering parameters in Table 4. Observed and calculated structure-factors are available from The Depository of Unpublished Data, CISTI, National Research Council, Ottawa, Ontario K1A 0S2, Canada.

Electron-Microprobe Analysis

The crystal used in the collection of the X-ray intensity data was extracted from a polished section. Analysis of the crystal prior to extraction showed the range of compositions indicated in Table 5. A BSE (back-scattered electron) image of the grain showed significant

TABLE 2. ATOMIC POSITIONS AND DISPLACEMENT FACTORS* FOR NIOBIUM-RICH CALCIOTANTITE FROM NYKÖPINGSGRUVAN

Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}	U_{eq}
Ta(1)	0.35938(5)	0.35938(5)	1/2	90(2)	90(2)	70(2)	1(1)	–1(1)	63(1)	76(1)
Ta(2)	1/3	2/3	1/4	91(2)	91(2)	62(3)	0	0	46(1)	82(2)
Ca	2/3	1/3	1/4	69(6)	69(6)	58(9)	0	0	35(3)	65(4)
O(1)	0.3757(7)	0.4304(8)	0.3440(3)	99(18)	121(17)	75(16)	25(14)	10(13)	57(14)	97(8)
O(2)	0.7532(9)	0.7532(9)	1/2	154(18)	154(18)	142(24)	–6(15)	6(15)	120(21)	131(10)
O(3)	1/3	2/3	0.5339(5)	75(17)	75(17)	127(26)	0	0	37(8)	92(12)

* $U \times 10^4$

TABLE 3. SELECTED INTERATOMIC DISTANCES (Å) FOR NIOBIUM-RICH CALCIOTANTITE FROM NYKÖPINGSGRUVAN

Ta(1)–O(1),a	1.958(4)	x2	*Ta*(2)–O(1),b,d,e,f,g	1.988(4) x6
Ta(1)–O(2)b,c	1.982(1)	x2		
Ta(1)–O(2)	2.452(6)		*Ca*–O(1),c,g,h,i,j	2.465(4) x6
Ta(1)–O(3),a	2.042(1)	x2	*Ca*–O(3)a,k	2.653(6) x2
<*Ta*(1)–O>	2.059		<*Ca*–O>	2.512

symmetry operators: a: $y, x, \bar{z}+1$; b: $\bar{x}+y, \bar{x}+1, z$; c: $\bar{y}+1, x-y, z$; d: $x, x-y+1, \bar{z}+½$; e: $\bar{y}+1, x-y+1, z$; f: $\bar{x}+y, y, \bar{z}+½$; g: $\bar{y}+1, \bar{x}+1, \bar{z}+½$; h: $\bar{x}+y+1, y, \bar{z}+½$; i: $\bar{x}+y+1, \bar{x}+1, z$; j: $x, x-y, \bar{z}+½$; k: $\bar{x}+1, \bar{y}+1, z-½$

TABLE 4. SITE POPULATIONS FOR *Ta* SITES (*apfu*)

Site	*Ta*	*Nb*	*Nb* / (*Nb* + *Ta*)
Ta(1)	2.094(6)	0.906(6)	0.302
Ta(2)	0.727(5)	0.273(5)	0.273

TABLE 5. CHEMICAL COMPOSITION (wt.%) AND UNIT FORMULA (*apfu*) FOR NIOBIUM-RICH CALCIOTANTITE FROM NYKÖPINGSGRUVAN

	Min. Ta*	Max. Ta*	Min. Ta**	Max. Ta**	Mean**
CaO	6.73	6.43	6.45	6.44	6.44
Ta_2O_5	71.72	79.69	73.58	74.53	74.09
Nb_2O_5	19.90	14.88	19.46	18.27	18.67
Sum	98.35	101.00	99.49	99.24	99.20
Ca	1.01	0.97	0.96	0.97	0.97
Ta	2.73	3.06	2.79	2.85	2.83
Nb	1.26	0.95	1.23	1.16	1.18
Sum	3.99	4.01	4.02	4.01	4.01

not detected: F, Na, Si, K, Ti, Mn, Fe, Sr, Sn, Sb, Cs, Ba, W, Pb, Bi, U

* Values from original large grain in polished section;
** Values from fragment extracted from polished section and used for X-ray intensity-data collection; mean is of four determinations.

variation in average atomic number (*i.e.*, Ta and Nb contents; Fig. 1, Smeds *et al.* 1999). We extracted the fragment used for structure work from the most homogeneous part of the crystal. Analysis was done with a Cameca SX–50 electron microprobe operating in wavelength-dispersion mode with an accelerating voltage of 15 kV, a specimen current of 20 nA, a beam size of 2 μm, and counting times on peak and background of 20 and 10 s, respectively. The following standards were used: $CaTa_4O_{11}$: Ca, Ta; $MnNb_2O_6$: Nb. Data were reduced using the $\phi(\rho Z)$ procedure of Pouchou & Pichoir (1985). The chemical composition is given in Table 5, together with the unit formula (calculated on the basis of 11 anions).

Coordination of the Cations

There is one *Ca* site in the structure of calciotantite; it is coordinated by eight O atoms with a <*Ca*–O> distance of 2.512 Å (Table 3). The six O atoms closest to *Ca* form a polyhedron that is intermediate between an octahedron and a triangular pyramid, and the two O atoms farther from *Ca* cap the two *trans* triangular faces of the triangular pyramid (Fig. 1a). There are two *Ta* sites, one coordinated by six O atoms in a distorted octahedral arrangement, and the other coordinated by seven O atoms in a distorted pentagonal dipyramidal arrangement (Fig. 1b). The two apical bonds to O(1) are the shortest bonds in this polyhedron, and the one long bond (2.45 Å) is to O(2), a meridional anion; however, these variations in bond length are in accord with the bond-valence requirements of the anions in the structure (Table 6).

Structure Topology

Calciotantite is a heteropolyhedral framework structure, but the polyhedron linkage is conveniently interpreted in terms of sheets parallel to (001). The $Ta(2)O_6$ and CaO_8 polyhedra occupy alternate vertices of a (planar) 6^3 net to form an open network of edge-sharing polyhedra of the form $[CaTaO_8]$ (Fig. 2a). The $Ta(1)O_7$ polyhedra share four (out of five) of their pentagonal

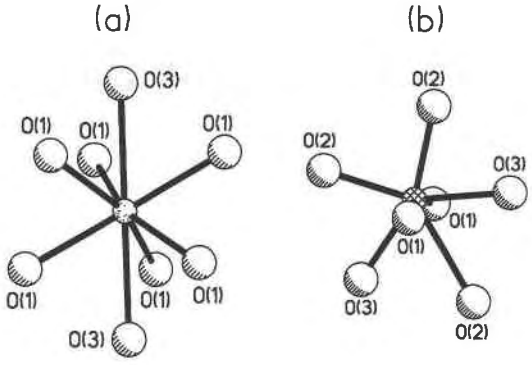

FIG. 1. The cation coordination in calciotantite: (a) *Ca;* (b) *Ta*(1); Ca: random-dot shaded; *Ta*(1): dash-shaded.

TABLE 6. BOND-VALENCE* TABLE (*vu*) FOR NIOBIUM-RICH CALCIOTANTITE FROM NYKÖPINGSGRUVAN

	Ta(1)	*Ta*(2)	*Ca*	Σ
O(1)	$0.90^{x2}\downarrow$	$0.83^{x6}\downarrow$	$0.26^{x6}\downarrow$	1.99
O(2)	$0.84^{x2}\downarrow\rightarrow$			1.92
	0.24			
O(3)	$0.71^{x2}\downarrow^{x3}\rightarrow$		$0.16^{x2}\downarrow$	2.29
Σ	5.14	4.98	1.88	

* Brown & Altermatt (1985)

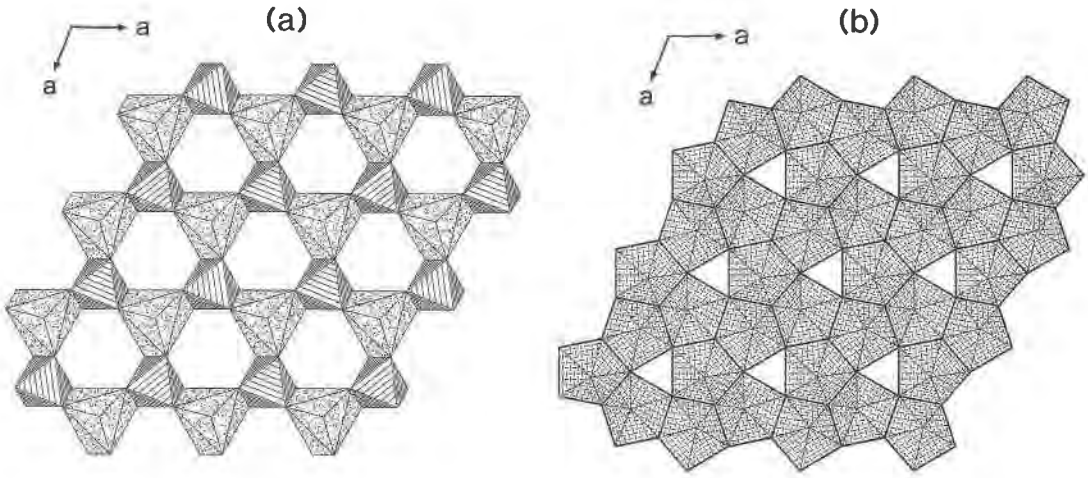

FIG. 2. (a) The $[CaTaO_8]$ sheet of edge-sharing $\{Ta(2)O_6\}$ octahedra and (CaO_8) polyhedra in the crystal structure of calciotantite; $\{Ta(2)O_6\}$ are line-shaded. (b) The $[Ta_3O_{11}]$ sheet of edge-and corner-sharing $\{Ta(1)O_7\}$ pentagonal dipyramids (dash-shaded) in the crystal structure of calciotantite. The pentagonal edges are shown by heavy lines; the legend is the same as in Figure 1.

edges to form a $[Ta_3O_{11}]$ sheet with a pattern of triangular holes [bounded by O(2) anions] at the vertices of a 6^3 net (Fig. 2b).

The two distinct sheets of polyhedra alternate along [001] to form a framework structure. Intersheet linkage occurs *via* the O(3) anions that constitute two of the five meridional anions in each pentagonal dipyramid of the $[Ta_3O_{11}]$ sheet, and the O(1) anions that are the apical bonds of the $Ta(1)O_7$ pentagonal dipyramids. The O(2) anions do not participate in the intersheet linkage. The

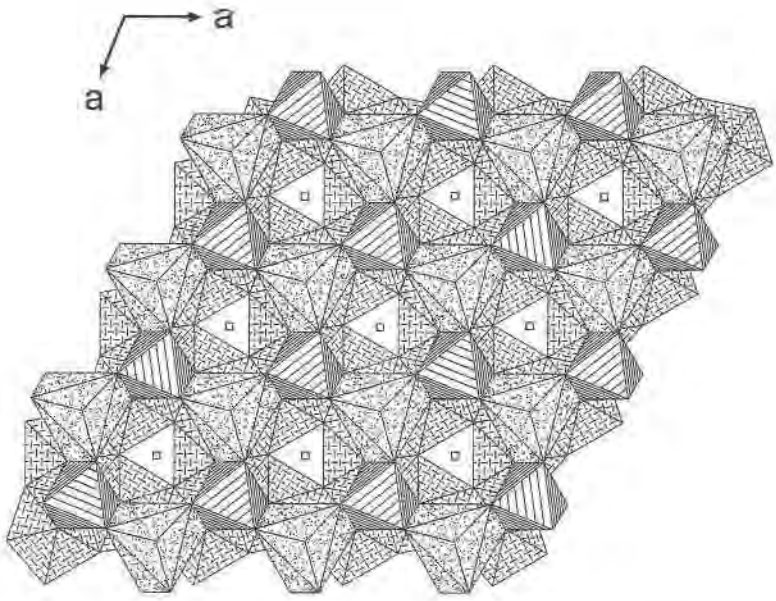

FIG. 3. Superposition of the $[Ta_3O_{11}]$ (lower) and $[CaTaO_8]$ (upper) sheets in the crystal structure of calciotantite. The legend is the same as in Figure 1; □ denotes the vacant octahedrally coordinated site discussed in the text

hexagonal holes of the $[CaTaO_8]$ sheet align with the triangular holes of the $[Ta_3O_{11}]$ sheet to form narrow tunnels extending along [001] (Fig. 3). Centered at 0, 0, ¼ is a flattened octahedral cavity with a center–vertex distance of 2.77 Å (□–O(1) × 6); this is a site that potentially might be involved in a solid-solution relation.

Related Structures

Natrotantite

The structure of calciotantite, $CaTa_4O_{11}$, is closely related to the structure of natrotantite, $Na_2Ta_4O_{11}$ (Ercit *et al.* 1985). Comparison of the cell dimensions (Table 7) shows that both have similar *a* dimensions on a hexagonal cell, whereas natrotantite has a *c* dimension close to three times the *c* dimension of calciotantite (36.62 *versus* 36.84 Å). The $[Ta_3O_{11}]$ sheets have identical topologies in these two structures, but the interleaved sheets, $[CaTaO_8]$ in calciotantite and $[Na_2TaO_8]$ in natrotantite, are somewhat different. In calciotantite, each $Ta(2)O_6$ octahedron shares edges with three CaO_8 polyhedra and three vacant polyhedra, $\square O_6$ (Fig. 2a). In natrotantite, each $Ta(2)O_6$ octahedron shares edges with six NaO_7 polyhedra. This difference means (1) that there are no holes in this sheet in natrotantite (Fig. 4), and (2) that the Na cation has the coordination number [7], compared to calciotantite, in which the Ca has [8]-coordination. Note that the $[CaTaO_8]$ and $[Na_2TaO_8]$ sheets are the same if they are written as $[\square CaTaO_8]$ and $[Na_2TaO_8]$. It is the change in coordination number of *Na* that is related to the difference in stacking sequence of the sheets in the two structures. In calciotantite, the *trans* nature of the linking O(3) anions means that next-nearest-neighbor sheets line up such that narrow but continuous tunnels penetrate the structure along [001]. In natrotantite, the "capping" anion occurs only on one end of the NaO_7 polyhedron (accounting for the change in coordination number from [8] to [7]). Thus in projection (Fig. 4), adjacent NaO_7 polyhedra are capped at the opposite ends (relative to the *c* axis) of the polyhedra.

TABLE 7 CELL DIMENSIONS AND SPACE GROUPS FOR NIOBIUM-RICH CALCIOTANTITE FROM NYKÖPINGSGRUVAN AND NATROTANTITE*

	Calciotantite	Natrotantite
a (Å)	6.2261(6)	6.2092(1)
c	12.280(1)	36.619(1)
V (Å^3)	412.18(8)	1222.67(4)
Sp Gr	$P6_322$	$R\bar{3}c$

* data from Ercit *et al.* (1985)

U-minerals

The $[Ta_3O_{11}]$ sheet in calciotantite and natrotantite is topologically identical to the $[(UO_2)_3\phi_5]$ sheet (ϕ = O,OH) in billietite, protasite, becquerelite and α–U_3O_8 (Burns *et al.* 1996).

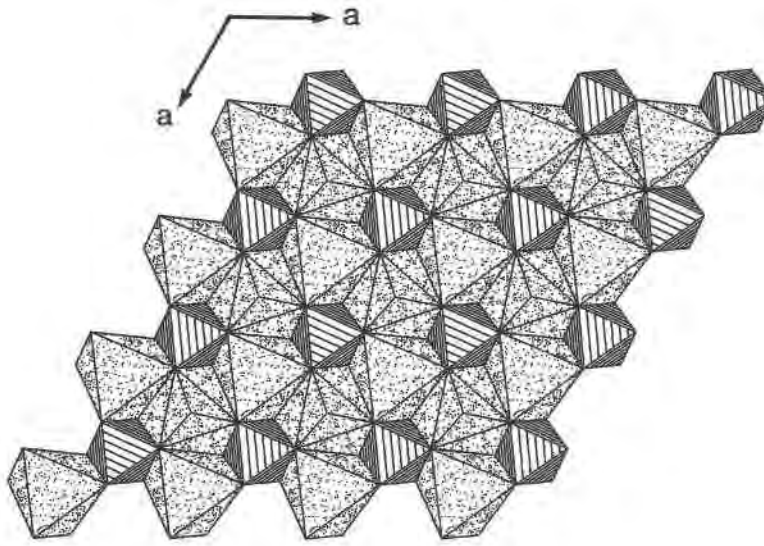

FIG. 4. The $[Na_2TaO_8]$ sheet in $Na_2Ta_4O_{11}$; (NaO_7) polyhedra are random dot-shaded, and $\{Ta(2)O_6\}$ octahedra are line-shaded. Note that adjacent (NaO_7) polyhedra are capped along $+c$ and $-c$, respectively.

Ta–Nb Order in Calciotantite

The formal charge (5^+) and empirical radii ($^{[6]}r$ = 0.64, $^{[7]}r$ = 0.69 Å, Shannon 1976) of Ta and Nb are the same, and (Ta,Nb)-oxide minerals show little or no evidence of order involving Ta and Nb. However, most minerals have Ta and Nb in octahedral coordination. The occurrence of both octahedral and pentagonal bipyramidal coordinations in calciotantite with significant amounts of both Ta and Nb provide an ideal opportunity to detect any crystal-chemical differences in the behavior of these two species.

As indicated in Table 4, Nb is preferentially ordered at *Ta*(1) relative to *Ta*(2), contrasting with the result of Yamnova *et al.* (1988), who reported all Nb ordered at *Ta*(2) in a sample of Nb-poor calciotantite. Although the degree of order in Table 4 is only slight, it is distinct. Thus there is a slight difference detectable in the ordering behavior of Ta and Nb, although it is almost certainly too small to detect for sites with the same coordination number.

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