

**POLYAKOVITE-(Ce), (REE,Ca)₄ (Mg,Fe²⁺) (Cr³⁺,Fe³⁺)₂ (Ti,Nb)₂ Si₄ O₂₂,
A NEW METAMICT MINERAL SPECIES FROM THE ILMEN MOUNTAINS,
SOUTHERN URALS, RUSSIA: MINERAL DESCRIPTION AND CRYSTAL CHEMISTRY**

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ABSTRACT

Polyakovite-(Ce), ideally $REE_4 \text{Mg Cr}^{3+}_2 \text{Ti}_2 \text{Si}_4 \text{O}_{22}$, monoclinic, a 13.398(1), b 5.6974(5), c 11.042(1) Å, β 100.539(2)°, V 828.6(2) Å³, $a:b:c = 2.3516:1:1.9381$, space group $C2/m$, $Z = 2$, is a new mineral species from the Ilmen Mountains, southern Urals, Russia. It is of hydrothermal origin; associated minerals are calcite, dolomite, fluororichterite, phlogopite, forsterite, monazite-(Ce), clinohumite, chromite and davidite-(Ce). It occurs as irregular grains up to 3 cm in diameter and euhedral crystals elongate along [010] (up to 2 mm) and flattened on {001}. The main forms are $a\{100\}$, $c\{001\}$, $t\{201\}$, $\kappa\{201\}$, $m\{110\}$, $o\{111\}$, $n\{\bar{1}11\}$, $p\{112\}$, $\rho\{\bar{1}12\}$ and $x\{\bar{3}02\}$. It is translucent in thin fragments, black with a brown streak, has a vitreous luster, and does not fluoresce under ultraviolet light. Polyakovite-(Ce) has a Mohs hardness of 5½–6, is brittle with a conchoidal fracture, and has no cleavage or parting. The measured density is 4.75(7) g/cm³, D_{calc} is 5.05 g/cm³. It is highly metamict. The infrared absorption spectrum has maxima at 473, 970 and 1115 cm⁻¹. In reflected light, polyakovite-(Ce) is gray, non-pleochroic, isotropic, n in the range 1.931–1.935. The strongest five reflections in the X-ray powder-diffraction pattern of annealed material [d in Å(I)(hkl)] are: 2.715(100)(004), 3.18(50)(311), 5.44(40)(002), 3.15(40)($\bar{3}12$), and 2.849(40)(020). An electron-microprobe analysis gave SiO₂ 19.08, TiO₂ 9.49, FeO 1.09, Fe₂O₃ 4.30, MnO 0.05, MgO 2.61, CaO 1.06, Cr₂O₃ 7.42, Nb₂O₅ 3.98, ThO₂ 2.79, UO₂ 0.03, Y₂O₃ 0.38, Ce₂O₃ 24.24, La₂O₃ 15.94, Nd₂O₃ 4.76, Pr₂O₃ 2.01, Sm₂O₃ 0.38, H₂O 0.14, sum 99.75 wt.%; the Fe²⁺/Fe³⁺ ratio was derived by Mössbauer spectroscopy. The corresponding chemical formula is (Ce_{1.87}La_{1.24}Nd_{0.36}Pr_{0.16}Sm_{0.03}Y_{0.04}Ca_{0.24}Th_{0.13}) Σ 4.07 (Mg_{0.82}Fe²⁺_{0.19}Mn_{0.01}) Σ 1.02 (Cr_{1.24}Fe³⁺_{0.68}) Σ 1.92 (Ti_{1.51}Nb_{0.38}) Σ 1.89 Si_{4.03} O₂₂ [based on O = 22 apfu (atoms per formula unit)]. The mineral is named for Vladislav Olegovich Polyakov (1950–1993), who contributed greatly to our knowledge of the mineralogy of the Urals.

The crystal structure of polyakovite-(Ce) (annealed single crystal) has been solved by direct methods and refined by least-squares to an R value of 4.6% using 1074 unique observed ($|F_o| > 4\sigma F$) reflections collected with a single-crystal diffractometer fitted with a CCD detector and MoK α X-radiation. Polyakovite-(Ce) is a sorosilicate of Cr³⁺, Ti⁴⁺, Fe²⁺ and REE³⁺, and the first titanosilicate where Cr³⁺ is a species-forming element. In the crystal structure of polyakovite-(Ce), there are four octahedrally coordinated M sites: $M(1)$, 0.80 Mg + 0.20 Fe²⁺, $\langle M(1)\text{-O} \rangle$ 2.108 Å; $M(2)$, 1.52 Ti + 0.32 Nb + 0.16 □, $\langle M(2)\text{-O} \rangle$ 1.984 Å; $M(4)$ and $M(3)$ are occupied by 0.64 Cr³⁺ + 0.36 Fe³⁺, $\langle M(3,4)\text{-O} \rangle$ 1.995 Å. Two distinct Si sites are occupied solely by Si, and the resulting (SiO₄) tetrahedra share one vertex to form an [Si₂O₇] group: $\langle \text{Si-O} \rangle$ 1.614 Å, Si(1)–O–Si(2) 173.2(8)°. There are two types of chains of octahedra: (1) [$M(3)$ and $M(4)$] octahedra, and (2) $M(2)$ octahedra, parallel to the b axis, form together an octahedral layer parallel to (001). In a chain, every two octahedra have a common edge. $M(2\text{--}4)$ octahedral layers, $M(1)$

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octahedra, and $[\text{Si}_2\text{O}_7]$ groups form a three-dimensional framework. There are two *A* sites (CN = [8] and [10], respectively) in which Ce^{3+} is dominant over other REE^{3+} , Ca and Th^{4+} . Polyakovite-(Ce) differs from chevkinite-(Ce), $(\text{REE},\text{Ca})_4(\text{Fe}^{2+},\text{Mg})\text{Fe}^{3+}_2(\text{Ti},\text{Nb})_2\text{Si}_4\text{O}_{22}$, by the prevalence of Mg at the *M*(1) site and Cr^{3+} at the *M*(3) and *M*(4) sites.

Keywords: polyakovite-(Ce), new mineral species, titanosilicate, crystal structure, Mössbauer spectroscopy, Ilmen Mountains, Urals, Russia.

SOMMAIRE

Nous décrivons la polyakovite-(Ce), dont la formule idéale serait $\text{TR}_4\text{MgCr}^{3+}_2\text{Ti}_2\text{Si}_4\text{O}_{22}$, monoclinique, a 13.398(1), b 5.6974(5), c 11.042(1) Å, β 100.539(2)°, V 828.6(2) Å³, $a:b:c$ = 2.3516:1:1.9381, groupe spatial $C2/m$, Z = 2; il s'agit d'une nouvelle espèce minérale provenant des montagnes Ilmen, dans la partie sud de la chaîne des Ourales, en Russie. Elle a une origine hydrothermale; lui sont associées calcite, dolomite, fluororichtérite, phlogopite, forstérite, monazite-(Ce), clinohumite, chromite et davidite-(Ce). Elle se présente en grains irréguliers atteignant 3 cm de diamètre et en cristaux idiomorphes allongés selon [010] (jusqu'à 2 mm) et aplatis sur {001}. Les formes principales sont $a\{100\}$, $c\{001\}$, $t\{201\}$, $\kappa\{201\}$, $m\{110\}$, $o\{111\}$, $n\{\bar{1}11\}$, $p\{112\}$, $\rho\{\bar{1}12\}$ et $x\{302\}$. Elle est translucide en fragments minces, noire avec une rayure brune, possède un éclat vitreux, et ne montre aucune fluorescence en lumière ultraviolette. La polyakovite-(Ce) a une dureté de Mohs de 5½ à 6; elle est cassante avec une fracture conchoïdale, et ne montre aucun clivage ou plan de séparation. La densité mesurée est de 4.75(7) g/cm³, et D_{calc} est 5.05 g/cm³. Elle est complètement métamictite. Le spectre d'absorption infrarouge montre des maxima à 473, 970 et 1115 cm⁻¹. En lumière réfléchie, la polyakovite-(Ce) est grise, non pléochroïque, isotrope, avec n dans l'intervalle 1.931–1.935. Les cinq raies les plus intenses du spectre de diffraction X (méthode des poudres) du matériau reconstitué [d en Å(hkl)] sont: 2.715(100)(004), 3.18(50)(311), 5.44(40)(002), 3.15(40)($\bar{3}12$), et 2.849(40)(020). Une analyse à la microsonde électronique a donné SiO_2 19.08, TiO_2 9.49, FeO 1.09, Fe_2O_3 4.30, MnO 0.05, MgO 2.61, CaO 1.06, Cr_2O_3 7.42, Nb_2O_5 3.98, ThO_2 2.79, UO_2 0.03, Y_2O_3 0.38, Ce_2O_3 24.24, La_2O_3 15.94, Nd_2O_3 4.76, Pr_2O_3 2.01, Sm_2O_3 0.38, H_2O 0.14, somme 99.75% (poids); le rapport $\text{Fe}^{2+}:\text{Fe}^{3+}$ a été établi par spectroscopie de Mössbauer. La formule chimique correspondante, fondée sur 22 atomes d'oxygène par unité formulaire, est $(\text{Ce}_{1.87}\text{La}_{1.24}\text{Nd}_{0.36}\text{Pr}_{0.16}\text{Sm}_{0.03}\text{Y}_{0.04}\text{Ca}_{0.24}\text{Th}_{0.13})_{\Sigma 4.07}(\text{Mg}_{0.82}\text{Fe}^{2+}_{0.19}\text{Mn}_{0.01})_{\Sigma 1.02}(\text{Cr}_{1.24}\text{Fe}^{3+}_{0.68})_{\Sigma 1.92}(\text{Ti}_{1.51}\text{Nb}_{0.38})_{\Sigma 1.89}\text{Si}_{4.03}\text{O}_{22}$. Le nom honore Vladislav Olegovich Polyakov (1950–1993), qui a grandement contribué aux connaissances de la minéralogie des Ourales.

La structure cristalline de la polyakovite-(Ce), telle que déterminée sur un cristal unique reconstitué, a été résolue par méthodes directes et affinée par moindres carrés jusqu'à un résidu R de 4.6% en utilisant 1074 réflexions uniques observées ($|F_o| > 4\sigma F$) prélevées avec un diffractomètre muni d'un détecteur de type CCD et avec rayonnement $\text{MoK}\alpha$. La polyakovite-(Ce) est un sorosilicate de Cr^{3+} , Ti^{4+} , Fe^{2+} et TR^{3+} (TR : terres rares), le premier titanosilicate à contenir le Cr^{3+} comme constituant essentiel. Il y a quatre sites M à coordinence octaédrique: $M(1)$, 0.80 Mg + 0.20 Fe^{2+} , $\langle M(1)\text{-O} \rangle$ 2.108 Å; $M(2)$, 1.52 Ti + 0.32 Nb + 0.16 □, $\langle M(2)\text{-O} \rangle$ 1.984 Å; $M(4)$ et $M(3)$ contiennent 0.64 Cr^{3+} + 0.36 Fe^{3+} , $\langle M(3,4)\text{-O} \rangle$ 1.995 Å. Deux sites Si distincts contiennent exclusivement du Si, et les tétraèdres (SiO_4) partagent un atome d'oxygène pour former un groupe $[\text{Si}_2\text{O}_7]$: $\langle \text{Si-O} \rangle$ 1.607 Å, $\text{Si}(1)\text{-O-Si}(2)$ 173.2(8)°. Deux types de chaînes d'octaèdres sont présents: (1) des octaèdres [$M(3)$ et $M(4)$], et (2) des octaèdres $M(2)$, parallèles à l'axe b , forment ensemble un feuillet d'octaèdres parallèle à (001). Dans une chaîne, chaque paire d'octaèdres possède une arête commune. Les feuillets d'octaèdres $M(2\text{-}4)$, les octaèdres $M(1)$, et les groupes $[\text{Si}_2\text{O}_7]$ forment une trame tridimensionnelle. Il y a deux sites A , à coordinence [8] et [10], respectivement, dans lesquels le Ce^{3+} prédomine sur les autres terres rares, le Ca et le Th^{4+} . La polyakovite-(Ce) se distingue de la chevkinite-(Ce), $(\text{TR},\text{Ca})_4(\text{Fe}^{2+},\text{Mg})\text{Fe}^{3+}_2(\text{Ti},\text{Nb})_2\text{Si}_4\text{O}_{22}$, par l'importance de Mg au site $M(1)$ et du Cr^{3+} aux sites $M(3)$ et $M(4)$.

(Traduit par la Rédaction)

Mots-clés: polyakovite-(Ce), nouvelle espèce minérale, titanosilicate, structure cristalline, spectroscopie de Mössbauer, montagnes Ilmen, Ourales, Russie.

INTRODUCTION

In 1976, a (Cr,Mg)-dominant analogue of chevkinite was found by V.F. Zhdanov in a dolomite veinlet cross-cutting phlogopite–olivine rock in mine N97 (Ilmen Natural Reserve, Southern Urals, Russia), and described by Zhdanov *et al.* (1986). However, this material was not approved as a new mineral species at that time. We have re-investigated this material and present the results here. The new mineral and mineral name have been approved by the International Mineralogical Associa-

tion, Commission on New Minerals and Mineral Names. Polyakovite-(Ce) is named after Vladislav Olegovich Polyakov (1950–1993). Born in the city of Kirsanov in the Tambov district, central Russia, he worked at the Institute of Mineralogy, Ilmen Natural Reserve, contributed greatly to the mineralogy of the Urals, and initiated study of this mineral.

Cotype samples of polyakovite-(Ce) are deposited at the Museum of the Ilmen Natural Reserve (Miass, Chelyabinsk District, Russia) and the Fersman Mineralogical Museum (Moscow, Russia).

OCCURRENCE AND ASSOCIATED MINERALS

Polyakovite-(Ce) was found in a carbonatite vein in mine N97, Ilmen Natural Reserve (55°01' N, 60°11' E), in the southern Urals, Russia. The mine is located at the eastern edge of a North-Ilmen swamp at the contact between fenites and metasomatized ultramafic rocks. The latter consist of phlogopite–fluororichterite and phlogopite–olivine rocks exposed in a lenticular body that is concordant with the host amphibolites and fenites. The metasomatized ultramafic rocks are cut by a series of east–west fluororichterite – chondrodite – phlogopite – dolomite, forsterite – phlogopite – chondrodite – dolomite, and phlogopite – fluororichterite – dolomite – calcite veinlets containing accessory monazite-(Ce), fergusonite-(Y), aeschynite-(Ce), chromian spinel, chromian davidite-(Ce), calcite, dolomite, fluororichterite, phlogopite, forsterite, clinohumite and polyakovite-(Ce). A detailed geological description of the mine and a full description of the corresponding mineral associations can be found in Polyakov & Bazhenova (1989).

Polyakovite-(Ce) occurs as anhedral equant grains (usually 0.5–0.7 cm, but up to 2.5 cm in diameter) and as euhedral crystals elongate along [010] (up to 2 mm long), closely associated with dolomite and fluororichterite. Polyakovite-(Ce) grains may contain inclusions of fluororichterite, chromite and thorianite. Polyakovite-(Ce) is xenomorphic relative to fluororichterite and forsterite, but has common surfaces of simultaneous growth with dolomite. Rarely, polyakovite-(Ce) occurs in a carbonate-free phlogopite–fluororichterite rock (Zhdanov *et al.* 1986).

PHYSICAL AND OPTICAL PROPERTIES

Polyakovite-(Ce) is black with a brown streak, translucent in thin fragments, has a light brown streak, a vitreous luster, and does not fluoresce under ultraviolet light. Polyakovite-(Ce) has a Mohs hardness of 5½–6, and micro-indentation tests gave a mean of

874 kg/mm² with a range of 775–987 kg/mm² (VNH₂₀₀, PMT-3 device, NaCl calibrated). It is brittle with a conchoidal fracture and has no cleavage or parting. The measured density is 4.75(7) g/cm³ (microflotation), and the calculated density of the annealed material is 5.05 g/cm³; this difference is due to the fact that we cannot obtain a cell volume on polyakovite-(Ce) as it does not give a diffraction pattern. Polyakovite-(Ce) is highly metamict. In reflected light, polyakovite-(Ce) is gray, non-pleochroic, isotropic, 1.931 < *n* < 1.935; reflectance values are low (Table 1), and brown internal reflections are rarely observed.

Some grains of polyakovite-(Ce) have a fragmented appearance. An ideal monoclinic crystal (Fig. 1) has the main forms: *a*{100}, *c*{001}, *t*{201}, *κ*{ $\bar{2}$ 01}, *m*{110}, *o*{111}, *n*{ $\bar{1}$ 11}, *p*{112}, *ρ*{ $\bar{1}$ 12}, *x*{ $\bar{3}$ 02}; these forms are practically identical to those of chevkinite. From goniometry, the axial ratios for polyakovite-(Ce) are: *a*:*b*:*c* = 2.320:1:1.922, $\beta_{\text{meas}} \approx 100^\circ$. On annealing, polyakovite-(Ce) becomes crystalline. The DTA curve of polyakovite-(Ce) shows a relaxation exothermic effect at 830°C (Fig. 2). The IR spectrum of polyakovite-(Ce) (UR-20 spectrometer) is practically identical to spectra of chevkinite-group minerals, with three intense absorption bands at 473, 970 and 1115 cm⁻¹.

TABLE 1. REFLECTANCE VALUES FOR POLYAKOVITE-(Ce)*

λ (nm)	<i>R</i> (%)	λ (nm)	<i>R</i> (%)
420	10.9	560	10.9
440	11.3	580	10.8
460	11.3	600	10.6
480	11.1	620	10.5
500	10.9	640	10.5
520	10.9	660	10.2
540	10.9	680	10.2

* Si was used as a standard

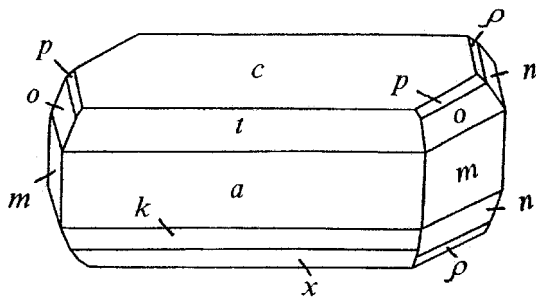


FIG. 1. Perspective view of an ideal crystal of polyakovite-(Ce) (taken from Zhdanov *et al.* 1986); the *b* axis is horizontal, and the *c* axis is vertical, both in the plane of the figure.

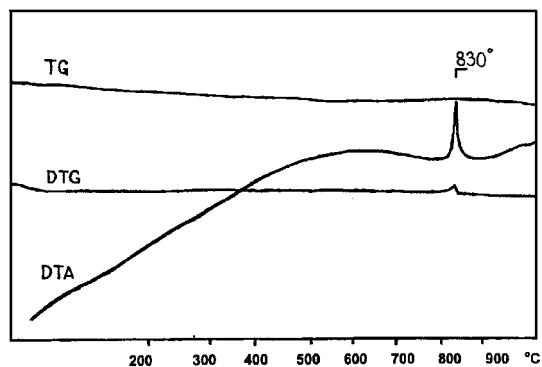


FIG. 2. Thermal analytical data for polyakovite-(Ce).

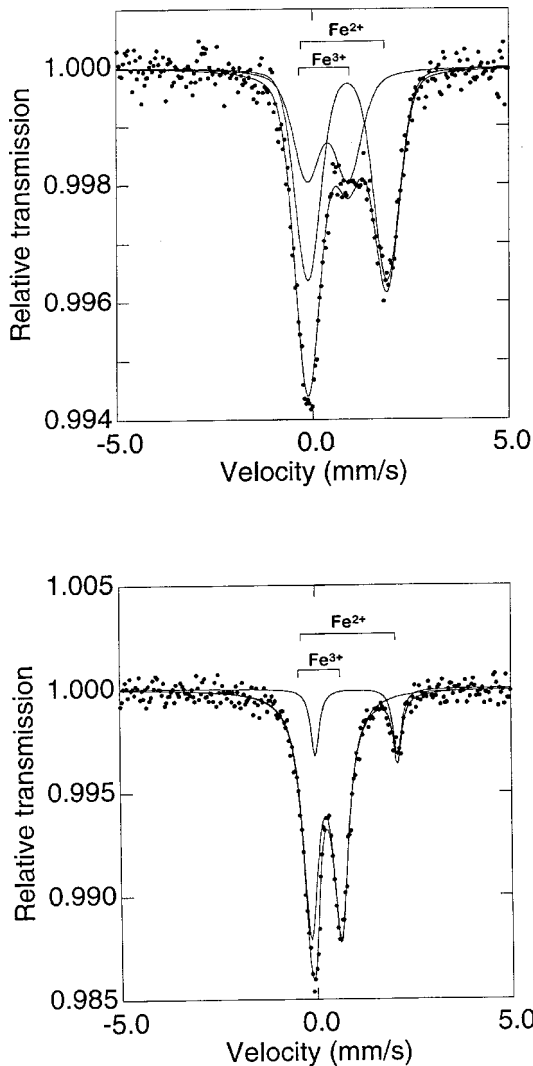


FIG. 3. Room-temperature Mössbauer spectra of (a) pre-annealed polyakovite-(Ce), and (b) annealed polyakovite-(Ce).

A Gladstone–Dale calculation gave a compatibility index of 0.042, good (on the basis of the measured density) and 0.099, poor (on the basis of the calculated density). A poor compatibility index is common for minerals in the metamict state.

CHEMICAL ANALYSIS

The chemical composition of polyakovite-(Ce) was determined by a combination of wet chemistry and electron-microprobe analysis (Table 2); JXA-50A electron

microprobe; 15 kV for Si, Ca, Mg; 20 kV for other elements; 30 nA. The following standards were used: diopside USNM 117733 (Si, Ca); ilmenite USNM 96189 (Ti, Fe); magnesiochromite USNM 117075 (Cr, Mg); Y_2O_3 (Y); (REE)P₅O₁₄ (Ce, La, Nd, Pr, Sm); ThO₂ (Th); LiNbO₃ (Nb); U_{Me} (U); Mn_{Me} (Mn) (USN standards from the Smithsonian Institution). Data were reduced using the “ZAF” correction (PUMA program). The ratio Fe²⁺/Fe³⁺ was measured by Mössbauer spectroscopy. The H₂O content (0.14 wt.%) was determined by the Penfield method. The empirical formula (Table 2) was calculated on the basis of 22 atoms of oxygen per formula unit, *apfu*. The ideal formula of polyakovite-(Ce) may be written as (REE³⁺,Ca)₄(Mg,Fe²⁺)(Cr,Fe³⁺)₂(Ti,Nb)₂Si₄O₂₂; the end-member formula is Ce³⁺₄MgCr³⁺₂Ti₂Si₄O₂₂.

MÖSSBAUER SPECTROSCOPY

Experimental

Two samples of polyakovite-(Ce) (pre-annealed and annealed) were gently ground in an agate mortar with acetone. The resulting powders were evenly distributed over a circular area on cellophane foil using water-soluble glue. Sample weights and diameter of the circular area were determined on the basis of the chemical composition such that (1) an ideal thickness of the

TABLE 2. CHEMICAL COMPOSITION (wt.%) AND EMPIRICAL FORMULA (*apfu*) OF POLYAKOVITE-(Ce)

	1	2	3	4	Average		
SiO ₂	18.50	19.40	19.38	19.02	19.08	Si	4.01
TiO ₂	9.76	9.36	9.24	9.61	9.49		
FeO	3.39	4.86	4.75	4.68	1.09*	Ti	1.50
Fe ₂ O ₃	2.41	—	—	—	4.30*	Nb	0.38
MnO	—	—	—	0.20	0.05	Σ	1.89
MgO	2.79	2.54	2.62	2.50	2.61		
CaO	1.39	0.95	0.95	0.95	1.06	Cr ³⁺	1.23
Cr ₂ O ₃	7.55	7.50	7.31	7.32	7.42	Fe ³⁺	0.68
Nb ₂ O ₅	2.36	4.56	4.56	4.43	3.98	Σ	1.91
ThO ₂	2.43	3.12	3.03	2.59	2.79		
UO ₂	—	0.03	0.02	0.07	0.03	Mg	0.82
Y ₂ O ₃	0.39	0.41	0.41	0.32	0.38	Fe ²⁺	0.19
Ce ₂ O ₃	26.16	23.65	23.93	23.20	24.24	Mn ²⁺	0.01
La ₂ O ₃	15.44	16.06	16.04	16.21	15.94	Σ	1.02
Nd ₂ O ₃	4.77	4.96	4.81	4.49	4.76		
Pr ₂ O ₃	1.77	1.98	2.21	2.08	2.01	Ce	1.87
Sm ₂ O ₃	0.34	0.43	0.43	0.34	0.38	La	1.24
H ₂ O*	—	—	—	—	0.14**	Nd	0.36
Total	99.45	99.81	99.69	98.01	99.75	Pr	0.15
						Sm	0.03
						Y	0.04
						Ca	0.24
						Th	0.13
						Σ	4.06

* Fe²⁺/Fe³⁺ ratio from Mössbauer spectroscopy;

** H₂O: Penfield method;

1: Wet chemistry; taken from Zhdanov *et al.* (1986); 2–4: EMPA.

sample was used, and (2) the sample diameter was maximized for the amount of sample available. The effective sample thickness was 2 mg Fe/cm², which is close to the ideal thickness of samples for these compositions (Long *et al.* 1983). Samples were run at room temperature (293 K) on a conventional transmission Mössbauer spectrometer.

Both the pre-annealed and annealed samples show two components corresponding to Fe²⁺ and Fe³⁺ (Fig. 3). The lines in the spectrum of the pre-annealed sample are relatively broad and were fit best with Voigt lineshapes, whereas the lines of the annealed sample are significantly narrower and gave the best fit with Lorentzian lineshapes. Component areas and widths of the doublets were constrained to be equal. The final parameters are given in Table 3.

RESULTS

The broad absorption lines in the pre-annealed sample (Fig. 3) indicate a wide range of environments for Fe²⁺ and Fe³⁺. It is not possible to assign them to specific crystallographic sites. In contrast, the absorption lines in the annealed sample are significantly narrower. In particular, Fe²⁺ has a linewidth close to the natural value, indicating that there is little variation in the electronic environment. One can unambiguously assign all Fe²⁺ in this sample to an octahedrally coordinated site, although it is not possible to identify which site. The center-shift value for Fe³⁺ lies within the range expected for octahedral coordination, so it is likely that octahedrally coordinated Fe³⁺ predominates. However, owing to the somewhat broad linewidth, it is impossible

to rule out the possibility of a small amount of tetrahedrally coordinated Fe³⁺, although there is no evidence for it on statistical grounds. An Fe³⁺/ΣFe ratio of 46(8)% for the pre-annealed sample and 86(3)% for the annealed sample were determined unambiguously from the spectra, and indicate that the annealed sample is more oxidized than the pre-annealed sample.

X-RAY POWDER DIFFRACTION

The highly metamict natural polyakovite-(Ce) crystals did not give an X-ray-diffraction pattern. Polyakovite-(Ce) was annealed for 3 h up to 1000°C in a helium atmosphere. X-ray powder-diffraction patterns for polyakovite-(Ce) annealed in a helium atmosphere, and central parts of large grains (more than 8 mm in diameter) annealed in air, are identical (Table 4).

TABLE 3. MÖSSBAUER PARAMETERS FOR POLYAKOVITE-(Ce)

	pre-annealed	annealed
Fe²⁺		
centre shift (mm/s) (rel. to α-Fe)	1.00(10)	1.158(8)
quadrupole splitting (mm/s)	2.02(21)	2.11(2)
Gaussian standard deviation (mm/s)	0.28(2)	—
Lorentzian linewidth (mm/s)	0.195*	0.25(2)
Fe³⁺		
centre shift (mm/s) (rel. to α-Fe)	0.50(22)	0.356(5)
quadrupole splitting (mm/s)	1.05(43)	0.775(6)
Gaussian standard deviation (mm/s)	0.23(6)	—
Lorentzian linewidth (mm/s)	0.44(24)	0.479(9)

*fixed to the natural line-width of ⁵⁷Fe

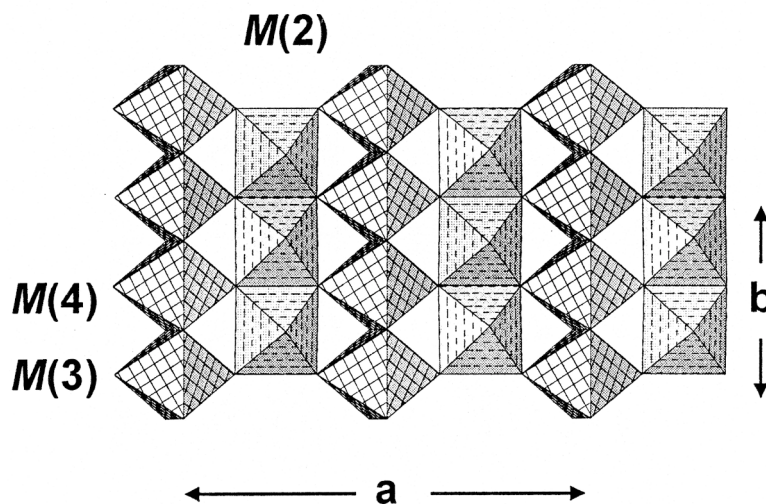


FIG. 4. The layer of octahedra in polyakovite-(Ce) projected onto (001). The M(2) octahedra are shaded by broken lines; the M(3) and M(4) octahedra, which have identical site-populations, are shaded by a 4⁴ net.

TABLE 4. POWDER X-RAY DIFFRACTION PATTERN FOR ANNEALED POLYAKOVITE-(Ce)

l_{obs}	$d_{\text{obs.}} (\text{\AA})$	$d_{\text{calc.}} (\text{\AA})$	hkl	l_{obs}	$d_{\text{obs.}} (\text{\AA})$	$d_{\text{calc.}} (\text{\AA})$	hkl
28	10.90	10.855	001	15	2.522	2.522	022
40	5.44	5.428	002	45	2.160	2.163	421
25	4.85	4.851	$\bar{1}11$	6	2.087	2.090	422
25	4.59	4.583	111	6	2.067	2.067	421
35	3.62	3.618	003	28	1.962	1.965	024
32	3.48	3.478	310	"	"	1.963	514
50	3.18	3.181	311	20	1.793	1.795	424
40	3.15	3.142	312	"	"	1.792	712
35	3.12	3.082	$\bar{1}13$	18	1.739	1.741	603
28	3.08	3.076	402	"	"	"	331
22	3.01	3.002	401	"	"	1.739	620
15	2.97	2.952	203	15	1.730	1.732	315
10	2.881	2.879	113	"	"	1.730	622
40	2.849	2.849	020	12	1.672	1.674	801
30	2.752	2.753	312	"	"	1.673	116
100	2.715	2.714	004	10	1.619	1.617	333
8	2.619	2.615	220				

*DRON-2 diffractometer, FeK α X-radiation, graphite monochromator;
Refined cell-dimensions: a 13.395(7), b 5.698(3), c 11.040(7) \AA , β 100.55(6) $^\circ$,
 V 828.5(6) \AA^3

SINGLE-CRYSTAL X-RAY DIFFRACTION

Collection of X-ray data and crystal-structure refinement

A single crystal for structure study was selected from the inner part of a sample annealed in air. Single-crystal X-ray data for polyakovite-(Ce) were collected with a Siemens P4 diffractometer fitted with a CCD detector, using MoK α radiation and an irregular crystal measuring $0.025 \times 0.10 \times 0.10$ mm. Integrated intensities of 6742 reflections with $\bar{1}8 \leq h \leq 18$, $\bar{7} \leq k \leq 7$, $\bar{1}5 \leq l \leq 15$ were collected up to $2\theta = 60.12^\circ$ using 30 s per frame. The refined cell-parameters (Table 5) were obtained from 3918 reflections ($I > 10 \sigma I$). An empirical absorption correction (SADABS, Sheldrick 1998) was applied.

STRUCTURE SOLUTION AND REFINEMENT

The crystal structure of polyakovite-(Ce) was solved by direct methods; the SHELXTL 5.1 program was used for solution and refinement of the structure. Scattering factors for neutral atoms were taken from the International Tables for X-ray Crystallography (Ibers & Hamilton 1974). Details of the X-ray data collection and structure refinement are given in Table 5. The crystal structure of polyakovite-(Ce) was refined to an R index of 4.6% and a Gof of 1.233 for a total of 115 refined parameters. In spite of annealing the single crystal of polyakovite-(Ce), its metamict origin reveals itself in additional weak maxima in the difference-Fourier map calculated in the final stages of refinement. Two such maxima of about $4.5 e$ were identified in polyakovite-(Ce) and were included in the final refinement. These sites are characterized by short distances to O(7) and O(8): 0.912 and 1.390 \AA , respectively. Final atom parameters for polyakovite-(Ce) are given in Table 6, selected interatomic distances are presented in Table 7, refined octahedral site-scattering values are given in Table 8, and a bond-valence analysis is shown in Table 9. A table of structure factors may be obtained from The Depository of Unpublished Data, National Research Council, Ottawa, Ontario K1A 0S1, Canada.

DESCRIPTION OF THE STRUCTURE

Coordination of the cations

In the crystal structure of polyakovite-(Ce), there are two unique Si sites, each occupied by Si and surrounded by four O-atoms in a tetrahedral arrangement, with $\langle Si(1)-O \rangle = 1.608 \text{ \AA}$ and $\langle Si(2)-O \rangle = 1.619 \text{ \AA}$. There are four unique M sites: $M(1)$ 0.80 Mg + 0.20 Fe $^{2+}$, $M(2)$ 1.52 Ti + 0.32 Nb + 0.16 \square , $M(3) = M(4)$ 0.64 Cr + 0.36 Fe $^{3+}$. The correlation between $\langle M-O \rangle_{\text{calc}}$ and

TABLE 5. MISCELLANEOUS STRUCTURE-REFINEMENT INFORMATION FOR POLYAKOVITE-(Ce)

a (\AA)	13.398(1)	Crystal size (mm)	0.025 x 0.100 x 0.100
b	5.6974(5)	Radiation	MoK α
c	11.042(2)	2θ -range for data collection ($^\circ$)	60.12
β ($^\circ$)	100.539(2)	$R(\text{int})$ (%)	4.1
V (\AA^3)	828.6(2)	Reflections collected	6742
		$ F_o > 4\sigma F$	4160
Space group	$C2/m$	Unique reflections	1306
		$F_o > 4\sigma F$	1074
Z	2	Refinement method	Full-matrix least-squares on F^2 ; fixed weights proportional to $1/\sigma(F^2)$
Absorption coefficient (mm^{-1})	13.77	Goodness of fit on F^2	1.233
$F(000)$	1154.0	Final R index (%) [$F_o > 4\sigma F$]	4.6
		R index (%) (all data)	6.1
		wR_2	0.109

TABLE 6. FINAL ATOM PARAMETERS FOR POLYAKOVITE-(Ce)

	x	y	z	U_{eq}	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
A(1)	0.35606(5)	0	0.73540(7)	0.0152(2)	0.0122(3)	0.0194(4)	0.0139(4)	0	0.0020(3)	0
A(2)	0.07041	0	0.74116	0.0199(3)	0.0122(4)	0.0338(5)	0.0136(4)	0	0.0018(3)	0
M(1)	1/2	0	1/2	0.0169(13)	0.0200(22)	0.0143(22)	0.0161(22)	0	0.0022(15)	0
M(2)	1/4	1/4	0	0.0093(6)	0.0113(9)	0.0101(10)	0.0061(9)	0.0002(7)	0.0003(6)	0.0002(7)
M(3)	0	0	0	0.0120(9)	0.0135(14)	0.0156(15)	0.0067(13)	0	0.0017(9)	0
M(4)	1/2	0	0	0.0133(9)	0.0150(14)	0.0169(15)	0.0071(13)	0	-0.0003(9)	0
Si(1)	0.2022(3)	-1/2	0.7327(3)	0.0105(6)	0.0180(16)	0.0076(14)	0.0054(14)	0	0.0009(11)	0
Si(2)	0.3582(3)	-1/2	0.5452(3)	0.0123(7)	0.0128(14)	0.0135(16)	0.0103(15)	0	0.0017(12)	0
O(1)	0.4785(4)	0.252(1)	0.8726(5)	0.0108(11)	0.0114(25)	0.0107(29)	0.0097(26)	0.0005(23)	0.0004(20)	-0.0005(22)
O(2)	0.1472(6)	0	0.9782(8)	0.0109(16)	0.0138(37)	0.0090(40)	0.0107(40)	0	0.0042(31)	0
O(3)	0.1888(7)	0	0.5982(8)	0.0146(18)	0.0193(42)	0.0189(47)	0.0046(36)	0	-0.0004(31)	0
O(4)	0.3495(6)	0	0.9887(7)	0.0090(16)	0.0076(34)	0.0084(38)	0.0094(39)	0	-0.0026(29)	0
O(5)	0.4283(6)	0.2689(3)	0.5915(6)	0.0254(16)	0.0340(38)	0.0247(39)	0.0159(33)	0.0021(29)	0.0003(28)	0.0120(32)
O(6)	0.2279(5)	-0.736(1)	0.8147(5)	0.0132(12)	0.0220(29)	0.0109(29)	0.0060(25)	-0.0003(23)	0.0010(21)	0.0028(24)
O(7)	0.0869(8)	-1/2	0.669(1)	0.0302(27)	0.0201(48)	0.0576(83)	0.0117(47)	0	-0.0005(38)	0
O(8)	0.2750(9)	-1/2	0.632(1)	0.0354(30)	0.0345(59)	0.0619(90)	0.0151(50)	0	0.0185(44)	0

TABLE 7. SELECTED INTERATOMIC DISTANCES (Å) AND ANGLES (°) IN POLYAKOVITE-(Ce)

A(1)–O(1)	x2	2.477(6)	A(2)–O(1)	x2	2.505(6)
A(1)–O(3)		2.465(8)	A(2)–O(2)		2.631(8)
A(1)–O(4)		2.814(8)	A(2)–O(3)		2.434(9)
A(1)–O(5)	x2	2.523(8)	A(2)–O(5)	x2	2.634(7)
A(1)–O(6)	x2	<u>2.554(6)</u>	A(2)–O(6)	x2	2.597(6)
<A(1)–O>		2.548	A(2)–O(7)	x2	<u>2.977(3)</u>
			<A(2)–O>		2.649
M(1)–O(5)	x4	2.156(7)	M(2)–O(2)	x2	1.966(6)
M(1)–O(7)	x2	<u>2.011(10)</u>	M(2)–O(4)	x2	1.970(5)
<M(1)–O>		2.108	M(2)–O(6)	x2	<u>2.015(6)</u>
			<M(2)–O>		1.984
M(3)–O(1)	x4	1.978(6)	M(4)–O(1)	x4	1.994(6)
M(3)–O(2)	x2	<u>2.030(8)</u>	M(4)–O(4)	x2	<u>1.998(8)</u>
<M(3)–O>		1.995	<M(4)–O>		1.995
Si(1)–O(6)		1.624(6)	O(6)–Si(1)–O(6)'		112.0(5)
Si(1)–O(7)	x2	1.575(10)	O(6)–Si(1)–O(7)	x2	109.9(3)
Si(1)–O(8)		<u>1.609(10)</u>	O(6)–Si(1)–O(8)	x2	107.0(3)
<Si(1)–O>		1.608	O(7)–Si(1)–O(8)		<u>111.1(6)</u>
			<O–Si(1)–O>		109.5
Si(2)–O(3)		1.593(9)	O(3)–Si(2)–O(5)	x2	114.1(3)
Si(2)–O(5)	x2	1.644(7)	O(3)–Si(2)–O(8)		113.8(6)
Si(2)–O(8)		<u>1.595(10)</u>	O(5)–Si(2)–O(5)'		106.5(6)
<Si(2)–O>		1.619	O(5)–Si(2)–O(8)	x2	<u>103.6(4)</u>
			<O–Si(2)–O>		109.3
			Si(1)–O(8)–Si(2)		173.2(8)

<M–O>_{obs} for all sites (Table 8) confirms the assigned site-populations. The Fe³⁺/ΣFe ratio of 78% derived from the SREF is lower than 86% determined by Mössbauer spectroscopy. However, the single crystal used for structure determination was taken from the inner part of an annealed sample and could well be less oxidized in view of the short duration of annealing. A similar composition of the M(3) and M(4) sites is

confirmed by the same <M–O> distances, 1.995 Å. There are two A sites (CN = [8] and [10]) occupied by REE³⁺, Ca and Th⁴⁺.

For the A sites, interatomic distances vary from 2.465 to 2.814 Å at A(1) and 2.434 to 2.977 Å at A(2). Among rare-earth elements (REE), Ce³⁺ is dominant. The structural formula derived from the X-ray site refinement and the chemical formula is (Ce_{1.86}La_{1.23}Nd_{0.35}Pr_{0.15}Sm_{0.01}Y_{0.04}Ca_{0.24}Th_{0.12})Σ_{4.00}(Mg_{0.80}Fe²⁺_{0.20})Σ_{1.00}(Cr_{1.28}Fe³⁺_{0.72})Σ_{2.00}(Ti_{1.52}Nb_{0.32}□_{0.16})Σ_{2.00}Si_{4.00}O_{22.00}.

Structure topology

The main building unit of the polyakovite-(Ce) structure is a layer of octahedra parallel to (001) (Fig. 4). The layer consists of two unique rutile-like chains of octahedra; the first chain is formed by M(2) octahedra, and the second chain is formed by M(3) and M(4) octahedra. In a chain, each octahedron shares two *trans* edges with adjacent octahedra. Chains of two types regularly alternate within the layer, and are connected through common vertices (Fig. 4). Intercalated between these layers are heteropolyhedral chains of [Si₂O₇] groups and M(1) octahedra (Fig. 5a) that extend in the *a* direction. Two outer vertices of one tetrahedron of the [Si₂O₇] group link to the apical vertices of M(2) octahedra (Figs. 5a, b) to form a fairly open heteropolyhedral framework. Regular arrangement of M(2) and M(3,4) octahedra within the layer results in relatively large cages that contain the A(1) and A(2) sites (CN = [8] and [10]) (Fig. 5a).

Related compounds

Polyakovite-(Ce) is the second structurally investigated specimen of a group of highly metamict minerals

TABLE 8. REFINED *M*-SITE-SCATTERING VALUES (*epfu*) AND ASSIGNED SITE-POPULATIONS (*apfu*) FOR POLYAKOVITE-(Ce)

	Refined site-scattering	Assigned site-population	Predicted site-scattering	$\langle M-O \rangle_{\text{calc}}^*$ (Å)	$\langle M-O \rangle_{\text{obs}}$ (Å)
<i>M</i> (1)	17.4(3)	0.80 Mg + 0.20 Fe ²⁺	14.80	2.112	2.108
<i>M</i> (2)	46.0(4)	1.52 Ti + 0.32 Nb + 0.16 □	46.16	2.003	1.984
<i>M</i> (3)	24.2(3)	0.64 Cr + 0.36 Fe ³⁺	24.72	2.021	1.995
<i>M</i> (4)	24.7(7)	0.64 Cr + 0.36 Fe ³⁺	24.72	2.021	1.995

* Calculated by summing the constituent ionic radii, values from Shannon (1976)

TABLE 9. BOND-VALENCE TABLE FOR POLYAKOVITE-(Ce)

	Si(1)	Si(2)	M(1)	M(2)	M(3)	M(4)	A(1)	A(2)	Σ
O(1)				0.52 ^{vd} ₁	0.50 ^{vd} ₁	0.43 ^{vd} ₁	0.39 ^{vd} ₁		1.84
O(2)				0.65 ^{vd} ₁	0.45 ^{vd} ₁			0.26	2.01
O(3)		1.09 ^{vd} ₁					0.44	0.48	2.01
O(4)				0.65 ^{vd} ₁		0.50 ^{vd} ₁	0.14		1.94
O(5)		0.94	0.29 ^{vd} ₁				0.36 ^{vd} ₁	0.27 ^{vd} ₁	1.86
O(6)	1.00 ^{vd} ₁			0.56 ^{vd} ₁			0.33 ^{vd} ₁	0.31 ^{vd} ₁	2.20
O(7)	1.13		0.43 ^{vd} ₁					0.11 ^{vd} ₁	1.78
O(8)	1.04	1.08							2.12
Σ	4.17	4.20	2.02	3.72	2.98	3.00	2.82	2.90	

Bond-valence curves (*vu*) from Brown & Altermatt (1985)

comprising perrierite itself, chevkinite-(Ce) and strontiochevkinite. In a broad sense, polyakovite-(Ce) and perrierite (Ce,La,Ca)₄ (Fe²⁺,Ca) (Ti,Al,Fe³⁺)₂ Ti₂ Si₄ O₂₂ (Bonatti & Gottardi 1950, Gottardi 1960, Calvo & Faggiani 1974) are structural dimorphs, neglecting the compositional difference at the *M* sites. The main difference between the two structure types is due to the linkage of *M*(2) octahedra and [Si₂O₇] groups (*cf.* Figs. 5, 6). After determination of the crystal structure of perrierite (Gottardi 1960), Peng & Bun (1964) proposed a model of the crystal structure of chevkinite (REE_{3.47}Ca_{0.74}Th_{0.01}) Fe²⁺ (Ti_{0.72}Fe³⁺_{0.44}Fe²⁺_{0.41}Al_{0.33}Mg_{0.08}) (O_{7.92}OH_{0.08}) [(Si_{1.855}Al_{0.145})O₇]₂. They predicted a structure based on analysis of the P(*uvw*) map and reported a space group of *C2/m*; a structure refinement has not yet been done for chevkinite. This model corresponds to the crystal structure determined for polyakovite-(Ce) in this work. Polyakovite-(Ce) differs from chevkinite-(Ce), (REE,Ca)₄ (Fe²⁺,Mg) Fe³⁺₂ (Ti,Nb)₂ Si₄ O₂₂, in the dominance of Cr³⁺ at the *M*(3) and *M*(4) sites, and Mg at the *M*(1) site. On the basis of X-ray powder-diffraction data, Haggerty & Mariano (1983) proposed the space group *P2₁/a* for the Sr-dominant analogue of chevkinite, strontiochevkinite: (Sr,La,Ce,La)₄ (Fe²⁺,Fe³⁺) (Ti,Zr)₄ Si₄ O₂₂. However, there are no structural data for this mineral. The space group *P2₁/a* was reported for a synthetic analogue of chevkinite, Co₂ Nd₄ Ti₃ Si₄ O₂₂ (Calvo & Faggiani 1974). This lower symmetry produces six unique *M* sites, in contrast to four *M* sites in polyakovite-(Ce) and three *M* sites in perrierite (Gottardi 1960). In general,

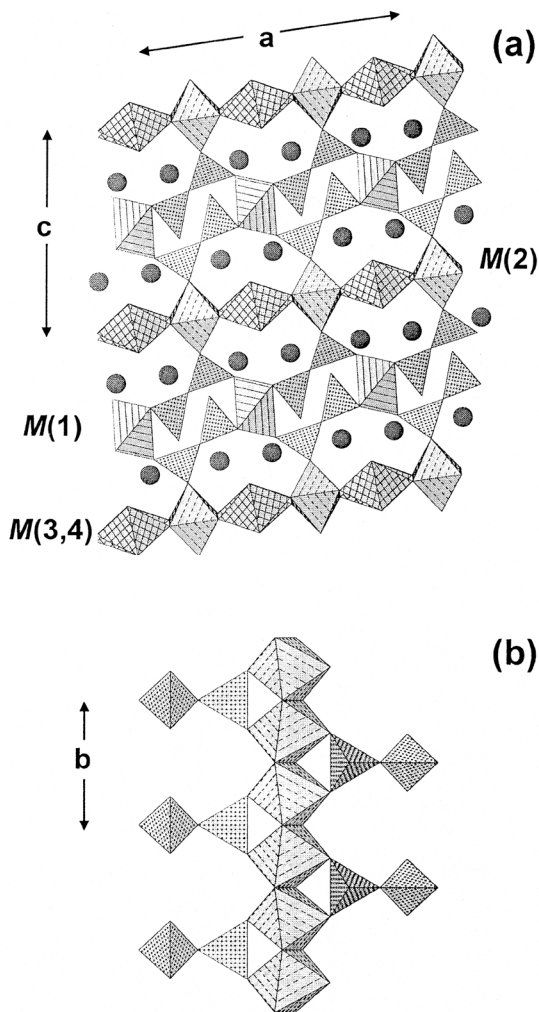


FIG. 5. Polyakovite-(Ce): (a) the crystal structure viewed down [010]; (b) the linkage of (SiO₄) tetrahedra and *M*(2) octahedra.

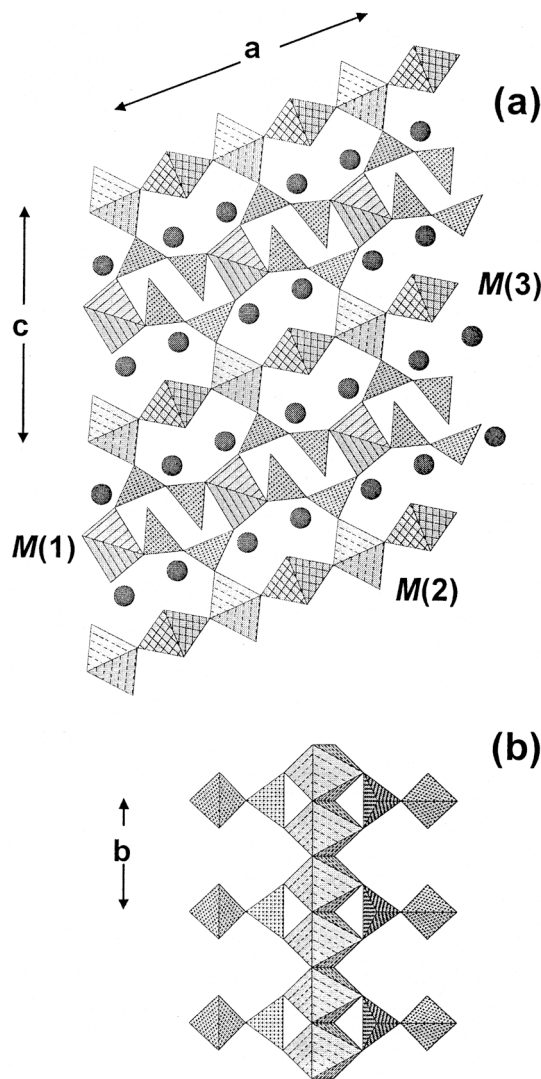


FIG. 6. Perrierite: (a) the crystal structure viewed down [010]; (b) the linkage of (SiO_4) tetrahedra and $M(2)$ octahedra.

the topologies of $\text{Co}_2\text{Nd}_4\text{Ti}_3\text{Si}_4\text{O}_{22}$, chevkinite (Peng & Bun 1964) and polyakovite-(Ce) are similar. Polyakovite-(Ce) is compared with related minerals in Table 10.

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TABLE 10. COMPARISON OF DATA FOR POLYAKOVITE-(Ce), CHEVKINITE-(Ce), STRONTIOCHEVKINITE AND PERRIERITE

	Polyakovite-(Ce) (ICDD 38-0443)	Chevkinite-(Ce) (ICDD 42-1394)	Strontiochevkinite (ICDD 38-0443)	Perrierite (ICDD 20-0260)
a (Å)	13.398(1)	13.395(5)	13.56	13.55
b	5.6974(5)	5.745(2)	5.70	5.63
c	11.042(1)	11.086(3)	11.10	11.70
β (°)	100.539(2)	100.65(3)	100.32	113.6
Space group	$C2/m$	$C2/m$	$P2_1/a$	$C2/m$
Z	2	2	2	2
Strongest lines in powder pattern	5.44, 40 4.85, 25 4.59, 25	5.45, 21 4.88, 29 4.608, 33		5.35, 10
d_{meas} (Å) (t)	3.48, 32	3.485, 38 3.472, 39		3.58, 10 3.52, 30
		3.18, 50 3.15, 40 3.01, 22	3.21, 10	3.11, 10 3.01, 30 2.957, 90
	2.861, 10 2.752, 30 2.715, 100	2.873, 49 2.760, 58 2.722, 100	2.85, 25	2.928, 100 2.819, 50 2.717, 20 2.679, 40 2.149, 30
Colour	Black	Black	Flesh-red	Black to brownish
Lustre	Resinous	Resinous	Submetallic	Resinous
D_{meas} (g/cm^3)	4.75	4.63	n.d.	4.3
D_{calc} (g/cm^3)	5.052	5.102	5.44	4.77
Hardness (Mohs)	5½–6	5–5½	~5	5½

Formulae: Polyakovite-(Ce): $(\text{REE}, \text{Ca})_4 (\text{Mg}, \text{Fe}^{2+}) (\text{Cr}, \text{Fe}^{3+})_2 (\text{Ti}, \text{Nb})_2 \text{Si}_4 \text{O}_{22}$;
 Chevkinite-(Ce): $(\text{REE}, \text{Ca})_4 (\text{Fe}^{2+}, \text{Mg}) \text{Fe}^{3+}_2 (\text{Ti}, \text{Nb})_2 \text{Si}_4 \text{O}_{22}$;
 Strontiochevkinite: $(\text{Sr}, [\text{La}, \text{Ce}]_{15} \text{Ca}_{23})_4 (\text{Fe}^{2+}, \text{Fe}^{3+}) (\text{Ti}, \text{Zr})_2 \text{Ti}_2 \text{Si}_4 \text{O}_{22}$;
 Perrierite: $(\text{Ce}, \text{La}, \text{Ca})_4 (\text{Fe}^{2+}, \text{Ca}) (\text{Ti}, \text{Al}, \text{Fe}^{3+})_2 \text{Ti}_2 \text{Si}_4 \text{O}_{22}$

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