




Article

Oxycalcimicrolite, $(\text{Ca},\text{Na})_2(\text{Ta},\text{Nb},\text{Ti})_2\text{O}_6(\text{O},\text{F})$, a new member of the microlite group (pyrochlore supergroup) from the Paleoproterozoic São João del Rei Pegmatite Province, Minas Gerais state, Brazil

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Abstract

Oxycalcimicrolite (IMA2019-110), $(\text{Ca},\text{Na})_2(\text{Ta},\text{Nb},\text{Ti})_2\text{O}_6(\text{O},\text{F})$, is a new member of microlite-group mineral found in the saprolite of the weathered Fumal pegmatite, located close to the city of Nazareno, Minas Gerais state, Brazil. It occurs as an accessory mineral associated with quartz, albite, microcline, muscovite, columbite-subgroup minerals, cassiterite, hematite, ilmenite, monazite-(Ce), xenotime-(Y), zircon, beryl, spinel, epidote and garnet-group minerals. Oxycalcimicrolite is found as octahedral crystals, occasionally modified to rhombododecahedra, ranging from 0.2 to 0.5 mm in size. The crystals are brownish-yellow to brownish-red and translucent, with white streak and vitreous to resinous lustre. The tenacity is brittle, with a Mohs hardness of 5–5½. Cleavage and parting are not observed; the fracture is conchoidal. Electron microprobe analysis, Raman and infrared spectroscopies and X-ray powder diffraction were applied to characterise this mineral. Oxycalcimicrolite is isotropic, $n_{\text{calc.}} = 2.037$, and the calculated density is 6.333 g/cm³. The composition is $(\text{Ca}_{1.57}\square_{0.26}\text{Na}_{0.06}\text{Sn}_{0.03}\text{Sr}_{0.03}\text{U}_{0.02}\text{Mn}_{0.02}\text{Fe}_{0.01}\text{Ce}_{0.01})_{\Sigma 2.00}(\text{Ta}_{1.79}\text{Nb}_{0.18}\text{Ti}_{0.03})_{\Sigma 2.00}\text{O}_{6.00}[\text{O}_{0.64}\text{F}_{0.19}\square_{0.17}]_{\Sigma 1.00}$ analysed by electron microprobe using wavelength dispersive spectrometry. The unit-cell parameters obtained by Pawley fitting from powder X-ray diffraction data are $a = 10.4325(4)$ Å and $V = 1135.46(14)$ Å³ with $Z = 8$.

Keywords: oxycalcimicrolite, pyrochlore supergroup, microlite group, Fumal pegmatite, São João del Rei Pegmatite Province

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Introduction

The nomenclature of the minerals belonging to the pyrochlore supergroup was revised by Atencio *et al.* (2010) and is approved by International Mineralogical Association Commission on New Minerals, Nomenclature and Classification (IMA–CNMNC). The new nomenclature uses the occupation of the A, B and Y sites and the general formula is written as $A_{2-m}B_2X_{6-w}Y_{1-m}$ where $m = 0$ to 1.7, $w = 0$ to 0.7 and $n = 0$ to 1 (Lumpkin and Ewing, 1992, 1995; Ercit and Robinson, 1994; Brugger *et al.*, 1997). The names are composed by two prefixes and one root name. The first prefix stands for the predominant ion of the dominant valence (or H₂O or vacancy '□') at the Y site while the second prefix represents the predominant cation of the dominant

valence (or H₂O or □) at the A site. The root names were defined according to the predominant cation of the dominant valence at the B site as: pyrochlore (if Nb⁵⁺ is the predominant cation of the dominant valence), microlite (Ta⁵⁺), roméite (Sb⁵⁺), betafite (Ti⁴⁺) or elsmoreite (W⁶⁺) (Atencio *et al.*, 2010). Hydrokenoralstonite and fluornatrocoulsellite were designated recently as unassigned members of the pyrochlore supergroup (Atencio *et al.*, 2017). In oxycalcimicrolite, the Y site is dominated by O²⁻, the A site by Ca²⁺ and the B site by Ta⁵⁺, presenting the ideal formula Ca₂Ta₂O₇. In natural crystals it is common to observe other elements occupying these sites, such that other authors have described similar samples but misclassified them as stibiomicrolite (Černý *et al.*, 2004) or microlite (Guastoni *et al.*, 2008).

Oxycalcimicrolite, $(\text{Ca},\square,\text{Na})_2\text{Ta}_2\text{O}_7$, is a new mineral of the microlite group of the pyrochlore supergroup (IMA2019-110, Menezes da Silva, *et al.*, 2020). It occurs in the Fumal pegmatite, which is located 18 km north of the city of Nazareno, Minas Gerais state, Brazil. The type material is deposited at Museu Nacional, Universidade Federal do Rio de Janeiro, Quinta da Boa Vista, s/n°, 20940-040, Rio de Janeiro, Brazil, under the

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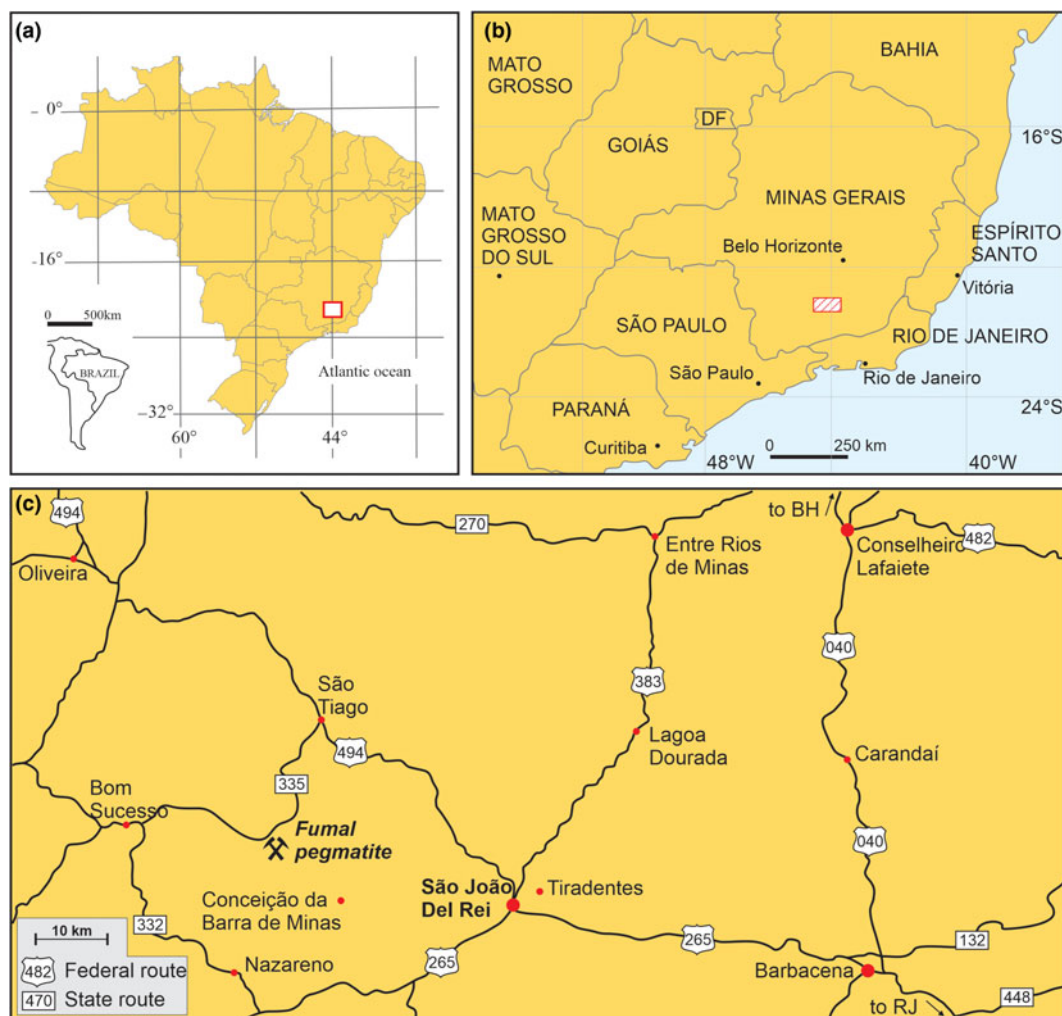


Fig. 1. Location map of the Fumal pegmatite: (a) map of Brazil, (b) detail showing location (hatched area) of (c) the position of the Fumal pegmatite in southern Minas Gerais state (modified from Faulstich, 2016).

registration code MN 7601-M. It is the first mineral described and deposited in the Museu Nacional after the September 2, 2018 fire.

Occurrence

Oxycalciumicrolite occurs as an accessory phase in the saprolite of the Fumal pegmatite ($21^{\circ}04'08.23''\text{S}$, $44^{\circ}33'59.63''\text{W}$), Nazareno, Minas Gerais, Brazil (Fig. 1). The pegmatite body is narrow (apparent thickness of 4 metres), deeply weathered and contains quartz, kaolinised albite and microcline, muscovite, columbite-subgroup minerals, cassiterite, hematite, ilmenite, monazite-(Ce), epidote-group minerals, xenotime-(Y), zircon, beryl, spinel and garnet-group minerals. The Fumal pegmatite belongs to the Sn-Ta-Nb-Li-rich São João del Rei Pegmatite Province (Pereira *et al.*, 2003); the evolution of this province is associated with the Paleoproterozoic granitoids of the Mineiro Belt (Ávila *et al.*, 2010, 2014; Teixeira *et al.*, 2015). Another important pegmatite of this province is the Volta Grande body (Lagache and Quéméneur, 1997; Alves *et al.*, 2019) which is currently mined for Li, Ta and Sn as well as feldspar for the ceramic industry. The oxycalciumicrolite characterised here was obtained through the concentration of heavy minerals from the pegmatite saprolite. The original material was concentrated using a pan to eliminate

excess quartz, feldspar and mica-group minerals (commonly 'biotite' and/or muscovite). The heavy-mineral concentrate was then separated using a dense liquid (methylene iodide S.D. = 3.32 kg/L) and ferrite hand magnet. A Frantz isodynamic separator with magnetic coil current of 0.1 A was then used to remove any residual ferromagnetic minerals and then ranging from 0.3 to 1.8 A, to separate the different para- and diamagnetic minerals. Pyrochlore-super-group minerals can be found in the concentrates separated from the 0.8 to 1.8 A range. The crystals chosen for characterisation are predominantly homogeneous and without evidence of weathering. Rarely some crystals were found to be compositionally altered to other species of microlite-group minerals, usually at the edges and close to fractures; these were not used for this study. Other crystals with different lustre and colours were also identified as microlite-group minerals in the same concentrate.

Appearance and physical properties

Oxycalciumicrolite occurs as octahedra, modified occasionally to rhombododecaedra with crystals generally ranging in size from 0.2 to 0.5 mm (Fig. 2). It is brownish-yellow to brownish-red in colour with a white streak. The mineral is translucent to

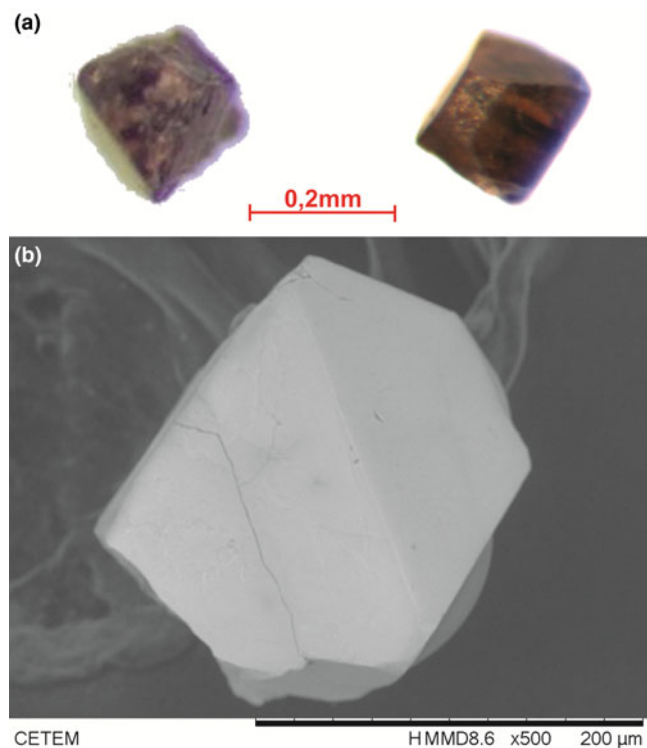


Fig. 2. Oxycalciumicrolite crystals, 0.2 mm in size from the same heavy minerals concentrate as the type specimen. (a) Stereomicroscope (b) back-scattered scanning electron and secondary electron composite image.

transparent with a vitreous to resinous lustre. It is non-fluorescent under longwave and shortwave ultraviolet light. It has a conchoidal fracture and no parting or cleavage was observed. The tenacity is brittle and the Mohs' hardness corresponds to 5–5½. The calculated density, based on the empirical formula and unit-cell parameters, is 6.333 g/cm³. The mineral is isotropic and the calculated refraction index, based on the empirical formula, is $n_{\text{calc.}} = 2.037$ using the Gladstone–Dale relationship $N = K_d + 1$ (K from Mandarino, 1981, d : density).

Composition

The composition of oxycalciumicrolite was determined using a JEOL JXA-8230 electron probe microanalyser (wavelength dispersive mode, 15 kV, 20 nA and 3 μm beam diameter). Analytical data from 14 spots are given in Table 1. The empirical formula calculated based on 2 cations at the *B* site is $(\text{Ca}_{1.57}\square_{0.26}\text{Na}_{0.06}\text{Sn}_{0.03}\text{Sr}_{0.03}\text{U}_{0.02}\text{Mn}_{0.02}\text{Fe}_{0.01}\text{Ce}_{0.01})_{\Sigma 2.00}(\text{Ta}_{1.79}\text{Nb}_{0.18}\text{Ti}_{0.03})_{\Sigma 2.00}\text{O}_{6.00}[\text{O}_{0.64}\text{F}_{0.19}\square_{0.17}]_{\Sigma 1.00}$ and the simplified formula is $\text{Ca}_2\text{Ta}_2\text{O}_7$. The mineral was named oxycalciumicrolite, based on nomenclature rules proposed by Atencio *et al.* (2010) and the dominant-valency rules (Bosi *et al.*, 2019), because O^{2-} predominates in the *Y* site, Ca^{2+} predominates in the *A* site and Ta^{5+} predominates in the *B* site.

A grain of oxycalciumicrolite was tested for reactions with water, nitric acid (70%), hydrochloric acid (37%), *aqua regia* and sulfuric acid (98%), all at room temperature. The grain did not react to any of these tests. When tested with hydrofluoric acid (48%), the mineral went through dissolution and showed a colour change from brownish to white.

Table 1. Chemical analyses of oxycalciumicrolite.

Constituent	Mean (wt.%)	Range (wt.%)	S.D.	Probe standard
Nb ₂ O ₅	4.12	1.95–7.42	1.59	Nb
Ta ₂ O ₅	75.77	72.32–78.63	2.24	Ta
TiO ₂	0.38	0.23–0.70	0.14	A-128
UO ₂	0.83	n.d.–5.67	1.49	UO ₂
ZrO ₂	0.03	n.d.–0.15	0.04	ZrSiO ₄
La ₂ O ₃	0.05	n.d.–0.24	0.07	LaPO ₄
Ce ₂ O ₃	0.18	0.15–0.29	0.04	CeO ₂
Yb ₂ O ₃	0.11	0.03–0.19	0.05	Yb
Sm ₂ O ₃	0.02	n.d.–0.11	0.03	SmPO ₄
SnO	0.94	0.44–2.03	0.42	SnO ₂
FeO	0.09	n.d.–0.29	0.08	Fe ₃ O ₄
CaO	15.89	14.44–16.44	0.49	Diopside
MnO	0.17	n.d.–0.33	0.13	Mn_Hort
SrO	0.51	0.36–0.74	0.13	Sr_AN
BaO	0.02	n.d.–0.09	0.03	Celsian
Na ₂ O	0.36	0.28–0.50	0.07	Jadeite
F	0.75	n.d.–1.71	0.43	CaF ₂
O = F	–0.31			
Total	99.91			

S.D. = standard deviation and n.d. = not detected.

Infrared data

The infrared data spectrum of oxycalciumicrolite was obtained from a powdered sample (6 mg) mixed, homogenised and pressed with anhydrous KBr (300 mg), using a PerkinElmer Spectrum 400 FT-IR/FT-NIR spectrometer, at the resolution of 16 cm⁻¹ and accumulation of 30 scans. A pure KBr disk was used as a background for the analyses.

The spectrum of oxycalciumicrolite (Fig. 3) shows bands in the range 400–700 cm⁻¹ and bands at 917 and 1000 cm⁻¹. The first group are associated with vibrations of the microlite type framework and the second one related to Ta–O octahedron vibrations (Andrade *et al.*, 2013a, b; Biagioni *et al.*, 2013; Andrade *et al.*, 2017; Guang *et al.*, 2017). No significant absorption attributed to H–O–H bending vibrations or O–H stretching vibrations, characteristically at 1600 to 1700 cm⁻¹ and 2900 to 3700 cm⁻¹, respectively, were observed. This is indicative of the absence of structural H₂O in oxycalciumicrolite.

Raman data

The Raman spectrum of oxycalciumicrolite was collected from a randomly oriented crystal using a Horiba Jobin Yvon LabRAM800 HR spectrometer coupled to Olympus BX41 microscope, and a thermoelectrically cooled CCD detector (at 203.15 K). Excitation was provided by a 632.8 nm wavelength He–Ne laser, while a 600 lines/mm diffraction grid ensured a spectral resolution of 1 cm⁻¹.

The Raman spectrum shows three bands between 100 and 1100 cm⁻¹ with vibrations at 295, 652 and 791 cm⁻¹ (Fig. 4). Based on previous studies (Glerup *et al.*, 2001; Arenas *et al.*, 2010), we assign the 295 and 652 cm⁻¹ bands to the *B*–*X* octahedral stretching and to the *X*–*B*–*X* bending, respectively. The 791 cm⁻¹ band could not be associated to any known vibrational mode of the pyrochlore supergroup, but it is possible that this band results from the combination, or overtone, of the 295 and 652 cm⁻¹ bands (Arenas *et al.*, 2010; Bahfenne and Frost, 2010).

X-ray diffraction

Powder X-ray data were obtained using a Bruker-AXS D8 Advance ECO equipment with an energy-discriminant LynxEye

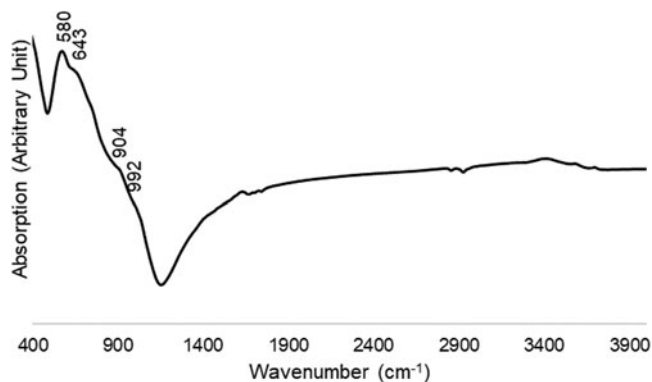


Fig. 3. Infrared spectrum of oxycalcimicrolite.

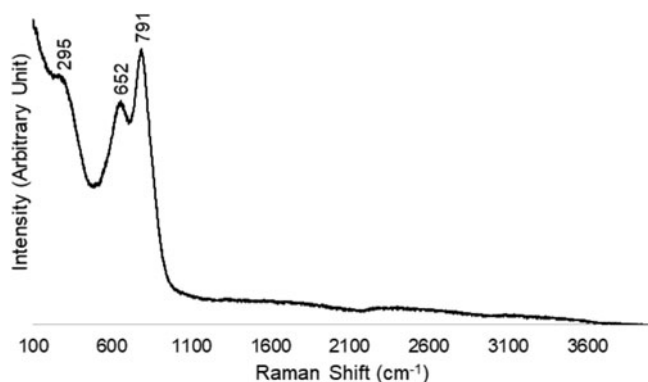


Fig. 4. Raman spectrum of oxycalcimicrolite.

XE detector, operating with unfiltered $\text{CuK}\alpha$ ($\lambda = 1.54056 \text{ \AA}$) radiation, collected from 4° to $105^\circ 2\theta$. Oxycalcimicrolite is cubic with space group $Fd\bar{3}m$. The powder X-ray lines and diffractogram are given in Table 2 and Fig. 5. Unit-cell parameters, obtained by the Pawley fitting from powder data are $a = 10.4325(4) \text{ \AA}$, $V = 1135.46(14) \text{ \AA}^3$ and $Z = 8$.

Discussion

According to the nomenclature and classification of the minerals belonging to the pyrochlore supergroup revised by Atencio *et al.* (2010), the mineral described in this study was named as oxycalcimicrolite, although other authors described and named similar minerals based on the original classification and nomenclature of the pyrochlore group (Hogarth, 1977). Černý *et al.* (2004) described a mineral named 'stibiomicrolite' in the Varutråsk pegmatite, northeastern Sweden, while Guastoni *et al.* (2008) identified a 'microlite' in the pegmatites of Vigizzo Valley (western Alps in Italy). Considering the available chemical data for these occurrences and following the classification scheme of Atencio *et al.* (2010), these minerals should be renamed as oxycalcimicrolite.

The small size of the crystal used for structure determination jeopardised its preparation for chemical analyses. Differences were observed between the electrons per formula unit (epfu) based on the chemical formula that satisfied the crystal-structure refinement and the product referring to the chemical analyses. As the composition and the crystal structure analyses could not be obtained from the same crystal, we chose not to present the data obtained by single-crystal diffraction.

Table 2. Powder X-ray diffraction data (d in \AA) for oxycalcimicrolite, indexed with $a = 10.4325(4) \text{ \AA}$, $V = 1135.46(14) \text{ \AA}^3$ and $Z = 8$.

$l_{\text{meas.}}$	$l_{\text{calc.}}$	$d_{\text{meas.}}$	$d_{\text{calc.}}$	h	k	l
89	64	6.023	5.9934	1	1	1
44	42	3.145	3.1294	3	1	1
100	100	3.011	2.9957	2	2	2
25	30	2.608	2.5947	4	0	0
6	7	2.393	2.3817	3	3	1
14	10	2.008	1.9978	3	3	3
48	34	1.8442	1.8350	4	4	0
11	9	1.7634	1.7547	5	3	1
10	4	1.5909	1.5829	5	3	3
33	25	1.5728	1.5648	6	2	2
8	6	1.5058	1.4982	4	4	4
6	4	1.4608	1.4536	7	1	1

The strongest lines are given in bold.

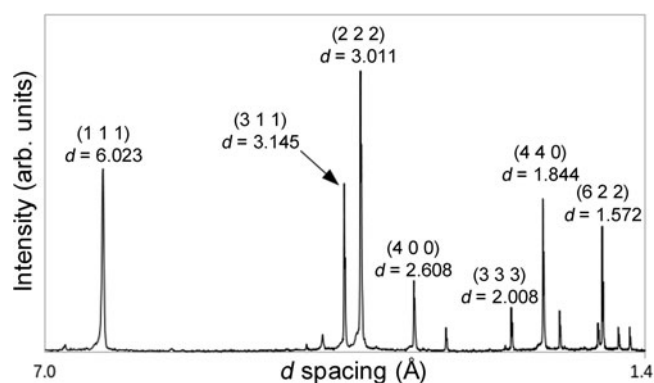


Fig. 5. Powder X-ray diffraction pattern of oxycalcimicrolite showing the principal Miller indices compatible with those of minerals of the pyrochlore supergroup.

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