

# Kenoplumbomicrolite, $(\text{Pb}, \square)_2\text{Ta}_2\text{O}_6[\square, (\text{OH}), \text{O}]$ , a new mineral from Ploskaya, Kola Peninsula, Russia

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[Received 15 May 2017; Accepted 16 October 2017; Associate Editor: Anthony Kampf]

## ABSTRACT

This study presents a complete characterization of kenoplumbomicrolite,  $(\text{Pb}, \square)_2\text{Ta}_2\text{O}_6[\square, (\text{OH}), \text{O}]$ , occurring in an amazonite pegmatite from Ploskaya Mountain, Western Keivy Massif, Kola Peninsula, Murmanskaja Oblast, Northern Region, Russia.

Kenoplumbomicrolite occurs in yellowish brown octahedral, cuboctahedral and massive crystals, up to 20 cm, has a white streak, a greasy lustre and is translucent. The Mohs hardness is ~6. Attempts to measure density (7.310–7.832 g/cm<sup>3</sup>) were affected by the ubiquitous presence of uraninite inclusions. Reflectance values were measured in air and immersed in oil. Kenoplumbomicrolite is optically isotropic. The empirical formula is  $(\text{Pb}_{1.30}\square_{0.30}\text{Ca}_{0.29}\text{Na}_{0.08}\text{U}_{0.03})_{\Sigma 2.00}(\text{Ta}_{0.82}\text{Nb}_{0.62}\text{Si}_{0.23}\text{Sn}_{0.15}\text{Ti}_{0.07}\text{Fe}_{0.10}^{3+}\text{Al}_{0.01})_{\Sigma 2.00}\text{O}_6[\square_{0.52}(\text{OH})_{0.25}\text{O}_{0.23}]_{\Sigma 1.00}$  (from the crystal used for the structural study) and  $(\text{Pb}_{1.33}\square_{0.66}\text{Mn}_{0.01})_{\Sigma 2.00}(\text{Ta}_{0.87}\text{Nb}_{0.72}\text{Sn}_{0.18}^{4+}\text{Fe}_{0.11}^{3+}\text{W}_{0.08}\text{Ti}_{0.04})_{\Sigma 2.00}\text{O}_6[\square_{0.80}(\text{OH})_{0.10}\text{O}_{0.10}]_{\Sigma 1.00}$  (average including additional fragments). The mineral is cubic, space group  $Fd\bar{3}m$ . The unit-cell parameters refined from powder X-ray diffraction data are  $a = 10.575(2)$  Å and  $V = 1182.6(8)$  Å<sup>3</sup>, which are in accord with those obtained previously from a single crystal of  $a = 10.571(1)$  Å,  $V = 1181.3(2)$  Å<sup>3</sup> and  $Z = 8$ . The mineral description and its name have been approved by the Commission on New Minerals, Nomenclature and Classification of the International Mineralogical Association (IMA2015-007a).

**KEYWORDS:** kenoplumbomicrolite, pyrochlore supergroup, uraninite, Ploskaya Mountain, Kola Peninsula, Russia.

## Introduction

THE general formula of the pyrochlore-supergroup minerals is  $A_2B_2X_6Y$ . The names are composed of two prefixes and one root name (identical to the name of the group). The first prefix refers to the dominant anion (or cation) of the dominant valence (or H<sub>2</sub>O or □) at the *Y* site. The second prefix refers

to the dominant cation of the dominant valence (or H<sub>2</sub>O or □) at the *A* site. The root names are elsmoreite, pyrochlore, microlite, roméite, betafite, ralstonite and coulsellite, depending on the nature of the predominant *B* cation and *X* anion (Atencio *et al.*, 2017). The dominant cations of the dominant valence at the *B* site are, respectively, W, Nb, Ta, Sb, Ti, Al and Mg. For elsmoreite, pyrochlore, microlite, roméite and betafite groups, the dominant anion of the dominant valence at the *X* site is O<sup>2-</sup>. For ralstonite and coulsellite groups, the dominant anion of the dominant valence at the *X* site is

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<https://doi.org/10.1180/minmag.2017.081.082>

F<sup>-</sup> (Atencio *et al.*, 2010, 2017; Christy and Atencio, 2013).

When the nomenclature scheme was approved (Atencio *et al.*, 2010), 20 names referring to existing minerals were listed, which needed a more complete description in order to be approved as valid species; among them is the mineral kenoplumbomicrolite, which involves  $Y = \square$  and  $A = \text{Pb}$ . Indeed, although several ‘plumbomicrolite’ occurrences are quoted in the literature, the status as a mineral species approved by the International Mineralogical Association (IMA) is still lacking (Atencio *et al.*, 2010).

Following the formula-calculation procedures, the ‘plumbomicrolite’ specimens previously described from Borborema Pegmatitic Province, Brazil (Beurlen *et al.*, 2005), from Urubu pegmatite, Itinga, Minas Gerais, Brazil (Uher *et al.*, 2008) and from Manono, Shaba, Democratic Republic of Congo (Uher *et al.*, 2008), should be named ‘kenomicrolite’, a still undescribed species that exhibits predominantly vacant  $A$  and  $Y$  sites.

Old chemical data refer to wet-chemical analyses and typically may represent a mixture of minerals, as indicated for data obtained by Voloshin *et al.* (1981) and Stepanov *et al.* (1982) for Ploskaya ‘plumbomicrolite’. The chemical data obtained by Stepanov *et al.* (1982) are similar to those presented in this work. It is not possible to decide if the sample studied by Chukanov (2014), from Ploskaya, is kenoplumbomicrolite, ‘oxyplumbomicrolite’, or ‘hydroxyplumbomicrolite’.

Safianikoff and van Wambeke (1961) obtained wet-chemical data for samples from Mumba, Kivu, Democratic Republic of Congo, which correspond to ‘kenomicrolite’, but microprobe data published by Ercit (1986) correspond to kenoplumbomicrolite. For additional ‘plumbomicrolite’ occurrences and data see Raade (2010).

With the aim of defining the status of IMA-approved species for one of these ‘orphan’ minerals of the pyrochlore supergroup, we describe here the new mineral kenoplumbomicrolite. This mineral has been studied already from a structural point of view by Bindi *et al.* (2006). For this reason, additional chemical data, physical properties and optical data were obtained from new fragments coming from the same locality.

Both the description and name (IMA2005-007a) were approved by the Commission on New Minerals, Nomenclature and Classification of the IMA. Type material is deposited in the collections of the Museu de Geociências, Instituto de Geociências, Universidade de São Paulo, Rua do Lago, 562,

05508-080 São Paulo, São Paulo, Brazil, registration number DR980.

## Occurrence

The mineral occurs as an accessory mineral in an amazonite pegmatite from Ploskaya Mountain (67° 37'60"N, 36°42'0"E), Western Keivy Massif, Kola Peninsula, Murmanskaja Oblast, Northern Region, Russia. The occurrence of ‘plumbomicrolite’ was described by Stepanov *et al.* (1982), who identified the mineral for the first time in 1975, in the quarry number 1 of the Ploskaya amazonite pegmatite vein on southwest slopes of the Keivy Range. The mineral in quarry number 1 was an irregularly shaped single-crystal grain of 15 cm × 15 cm × 12 cm with induced faces at the contact with biotite and microcline. At the biotite contact, it exhibits some octahedral and cubic faces. In 1976, several more ‘plumbomicrolite’ specimens were detected in the quarry number 5 of the same vein, as octahedra to 3 cm overgrowing cleavelandite flakes. The Mount Ploskaya vein that was being worked for amazonite was 270 m long. It has been traced downdip for 70 m and, in the central part, it had a thickness of 25 m. The vein is confined to the contact between gneiss and schist of the Keivy series of Proterozoic age and genetically related to an alkaline granite pluton. The contact zone consists of fine-grained albite and is succeeded nearer the centre by an indistinct zone of blocks of amazonite, as large as 2.5 m and partly replaced by blocks and saccharoidal or flaky aggregates of albite. The central pegmatite has a quartz core with galena pockets as large as 0.4 m, coarse biotite flakes and contains ‘cleavelandite’ inclusions.

Associated minerals are microcline (var. amazonite), albite (var. cleavelandite), quartz, biotite, ‘zinnwaldite’, anglesite, bastnäsite-(Ce), bismite, bismuth, bismuthinite, bismutite, cassiterite, caysichite-(Y), churchite-(Y), columbite-(Mn), emplectite, fergusonite-(Y), fluorite, gadolinite-(Y), gahnite, galena, hingganite-(Y), hingganite-(Yb), kainosite-(Y), kamphaugite-(Y), kasolite, keiviite-(Y), keiviite-(Yb), kuliokite-(Y), lanarkite, leadhillite, löllingite, incompletely studied pyrochlore-supergroup minerals, monazite-(Ce), pyromorphite, scotlandite, sillénite, sphalerite, tenerite-(Y), thalénite-(Y), thorite, uraninite, vyuntspakhkrite-(Y), wulfenite, xenotime-(Y), xenotime-(Yb) and zavaritskite. Kenoplumbomicrolite has uraninite inclusions.



FIG. 1. Kenoplumbomicrolite from Ploskaya, Kola Peninsula, Russia. The crystal size is 2 cm × 1.94 cm × 1.5 cm. Photo by Jasun McAvoy (<http://www.mineralman.com>). Authorized reproduction. This is not the type specimen.

The Ploskaya massif in the Kola Peninsula (Russia) is the type locality for five other minerals: hingganite-(Yb), keiviite-(Y), keiviite-(Yb), kulio-kite-(Y) and vyuntsphakhtite-(Y).

Both the specimen studied by Bindi *et al.* (2006) and the one used for the present study were supplied by the late Victor I. Stepanov, of the Fersman Museum, to Roy Kristiansen in 1979.

### Appearance and physical properties

Kenoplumbomicrolite (Fig. 1) forms yellowish brown octahedral, cuboctahedral and massive crystals, up to 20 cm (Anthony *et al.*, 1990). It exhibits a white streak, a greasy lustre and is translucent. It is non-fluorescent under long wavelength (365 nm) ultraviolet radiation. The Mohs hardness is ~6. The mineral is brittle, cleavage was not observed and the fracture is uneven. The measured density is 7.523 (7.310–7.832) g.cm<sup>-3</sup> (mean of five measurements using the pycnometer method), whereas the calculated density is 7.122 g.cm<sup>-3</sup> (for the crystal used for the structural study), using the empirical formula and X-ray data from single-crystal study. The discrepancy between the observed and calculated datum is due to the ubiquitous presence of inclusions of uraninite. Kenoplumbomicrolite is optically isotropic. Reflectance values (relative to a cubic zirconia standard) were measured in air and in an immersion oil with refractive index of 1.515 (Table 1 and Fig. 2). They were obtained using a J & M TIDAS diode array spectrophotometer attached to a Zeiss Axioplan reflected-light microscope on a homogeneous

area of a grain of kenoplumbomicrolite. No internal reflections were visible within the area of measurement.

Voloshin *et al.* (1981) presented the following data for kenoplumbomicrolite from Ploskaya (their sample 2): Density (meas.) = 7.69(7) g.cm<sup>-3</sup>;

TABLE 1. Reflectance values for kenoplumbomicrolite (*R* in %).

$\lambda$ (nm)	Air	Oil
400	20.35	8.13
420	19.77	7.41
440	19.29	7.05
460	18.77	6.74
<b>470</b>	<b>18.62</b>	<b>6.61</b>
480	18.48	6.49
500	18.18	6.22
520	17.90	6.04
540	17.69	5.95
<b>546</b>	<b>17.62</b>	<b>5.88</b>
560	17.46	5.71
580	17.30	5.67
<b>589</b>	<b>17.26</b>	<b>5.62</b>
600	17.22	5.56
620	17.08	5.39
640	16.93	5.36
<b>650</b>	<b>16.90</b>	<b>5.34</b>
660	16.88	5.32
680	16.80	5.30
700	16.75	5.28

The values required by the Commission on Ore Mineralogy are given in bold.

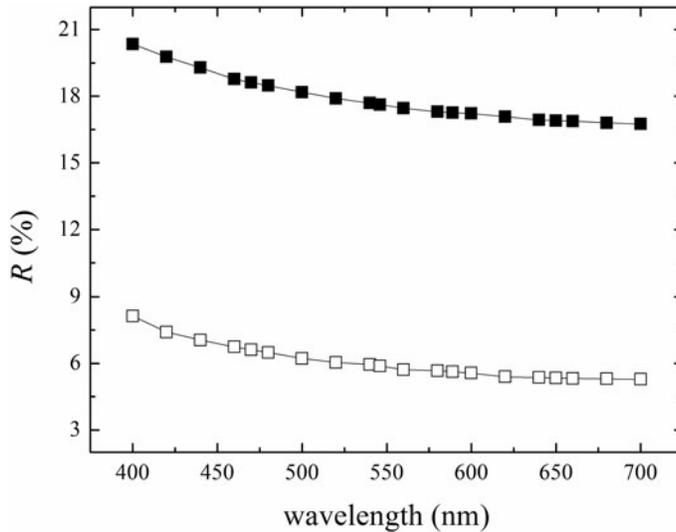


FIG. 2. Reflectance data for kenoplumbomicrolite. Filled and empty squares refer to measurements taken in air and oil, respectively.

VHN = 610 (40 g load); and  $R$  (nm, %) = 486, 21.6; 553, 21.2; 586, 21.7; 656, 20.1. The data from Voloshin *et al.* (1981) are for a crystal slightly more enriched in Pb, thus the observed density is slightly higher than that calculated for the crystal studied by Bindi *et al.* (2006).

#### Chemical data

Chemical data (Table 2) were obtained for two samples: (1) the crystal used for the structural study (Bindi *et al.*, 2006); and (2) additional fragments.

TABLE 2. Chemical data (wt.%) for kenoplumbomicrolite.

Constituent	Bindi <i>et al.</i> (2006)			This study		
	Mean	Range	Probe standard	Mean	Range	Probe standard
Na <sub>2</sub> O	0.37	0.32–0.46	albite			
CaO	2.51	2.18–2.63	kaersutite			
PbO	45.39	44.20–46.29	pure element	46.05	44.85–46.77	PbF <sub>2</sub>
UO <sub>2</sub>	1.24	0.99–1.45	pure element			
Ta <sub>2</sub> O <sub>5</sub>	28.58	27.21–28.83	pure element	29.95	29.78–30.23	pure element
Nb <sub>2</sub> O <sub>5</sub>	12.90	12.11–13.39	pure element	14.85	14.02–15.24	pure element
TiO <sub>2</sub>	0.84	0.74–0.90	rutile	0.49	0.22–0.74	pure element
SiO <sub>2</sub>	2.19	1.62–2.44	kaersutite			
SnO <sub>2</sub>	3.47	3.10–3.58	pure element			
Fe <sub>2</sub> O <sub>3</sub>	1.28	1.23–1.31	kaersutite	1.34	1.27–1.47	pure element
Al <sub>2</sub> O <sub>3</sub>	0.07	0.01–0.11	albite			
WO <sub>3</sub>				2.88	2.40–3.69	pure element
SnO <sub>2</sub>				4.27	4.00–4.76	pure element
MnO				0.10	n.d.–0.39	pure element
H <sub>2</sub> O	0.35					
Total	99.19			99.93		

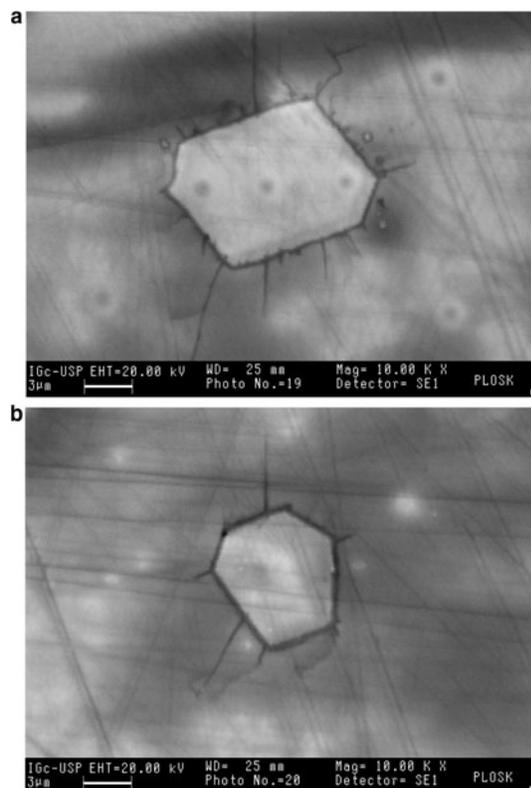


FIG. 3. (a,b) Back-scatter electron images showing uraninite inclusions in kenoplumbomicrolite.

Four chemical analyses were carried out on the crystal used for the structural study, using a Jeol JXA-8600 electron microprobe (wavelength dispersive spectroscopy mode, 15 kV, 20 nA and 30 s counting time). The crystal was found to be homogeneous within analytical error. H<sub>2</sub>O was calculated from the crystal structure. All Fe was assumed to be Fe<sup>3+</sup> and all Sn to be Sn<sup>4+</sup>. Mn, Sr, Sb, Ba, La, Ce, Pr, Nd, Tl and Th were sought but found to be below the detection limit. The empirical formula, based on 2 cations at the B site, is (Pb<sub>1.30</sub>□<sub>0.30</sub>Ca<sub>0.29</sub>Na<sub>0.08</sub>U<sub>0.03</sub>)<sub>Σ2.00</sub>(Ta<sub>0.82</sub>Nb<sub>0.62</sub>Si<sub>0.23</sub>Sn<sub>0.15</sub>Ti<sub>0.07</sub>Fe<sub>0.10</sub>Al<sub>0.01</sub>)<sub>Σ2.00</sub>O<sub>6</sub>[□<sub>0.52</sub>(OH)<sub>0.25</sub>O<sub>0.23</sub>]<sub>Σ1.00</sub>. The simplified formula is (Pb,□)<sub>2</sub>Ta<sub>2</sub>O<sub>6</sub>[□,(OH),O].

Seven chemical analyses were carried out on additional fragments (then used for density and reflectance measurements and powder X-ray diffraction investigations) using a LEO440 electron microscope equipped with Si-Li detector (EDS mode, 20 kV, 1.5 nA and 100 s counting time). The empirical formula, based on 2 cations at the B site, is (Pb<sub>1.33</sub>□<sub>0.66</sub>Mn<sub>0.01</sub>)<sub>Σ2.00</sub>(Ta<sub>0.87</sub>Nb<sub>0.72</sub>Sn<sub>0.18</sub>

Fe<sub>0.11</sub>W<sub>0.08</sub>Ti<sub>0.04</sub>)<sub>Σ2.00</sub>O<sub>6</sub>[□<sub>0.80</sub>(OH)<sub>0.10</sub>O<sub>0.10</sub>]<sub>Σ1.00</sub>. Mn was assumed as divalent. In this formula O and OH are in equal proportions at the Y site by analogy to the sample studied structurally. In the absence of H<sub>2</sub>O analysis [which is compensated by the crystal-structure determination study (Bindi *et al.*, 2006)], the Y site occupancy could be expressed in different ways, for example: [□<sub>0.70</sub>(OH)<sub>0.30</sub>] or [□<sub>0.85</sub>O<sub>0.15</sub>]. But, in any case, the vacancy will be dominant and the amount of O and/or (OH) pfu will be small.

TABLE 3. Chemical data (wt.%) for uraninite inclusions in kenoplumbomicrolite.

Constituent	Mean	Range	Probe standard
UO <sub>2</sub>	58.63	55.73–61.23	pure element
ThO <sub>2</sub>	21.69	18.64–23.07	ThO <sub>2</sub>
PbO	15.37	14.89–15.76	PbF <sub>2</sub>
Total	95.69		

TABLE 4. Powder X-ray diffraction data for kenoplumbomicrolite\*.

$d_{\text{obs.}}(\text{\AA})$	$I_{\text{obs}}$	$d_{\text{calc.}}(\text{\AA})^{**}$	$I_{\text{calc}}$	$hkl$
<b>3.050</b>	<b>100</b>	<b>3.0516</b>	<b>100</b>	2 2 2
<b>2.641</b>	<b>42</b>	<b>2.6428</b>	<b>37</b>	4 0 0
2.425	9	2.4252	1	3 3 1
		2.1578	1	4 2 2
2.033	6	2.0344	1	5 1 1
<b>1.869</b>	<b>26</b>	<b>1.8687</b>	<b>38</b>	4 4 0
<b>1.595</b>	<b>23</b>	<b>1.5936</b>	<b>37</b>	6 2 2
1.527	9	1.5258	10	4 4 4
		1.3214	5	8 0 0
		1.2126	13	6 6 2
		1.1819	12	8 4 0

\*Intensities and  $d_{\text{hkl}}$  values were calculated using the *XPOW* program software (Downs *et al.*, 1993) on the basis of the structural model given by Bindi *et al.* (2006); only reflections with  $I_{\text{calc}} > 1$  are listed. The strongest four reflections are given in bold.

\*\*Calculated for  $a = 10.571 \text{ \AA}$ .

Uraninite inclusions in kenoplumbomicrolite crystals from Ploskaya were already noted by Voloshin *et al.* (1981). As is evident from Fig. 3, radiating cracks can be observed around uraninite. They can be interpreted as the result of microfracturing caused by radioactivity. Uraninite usually undergoes intense self-irradiation which can lead to amorphization, which, in turn, induces a volume increase and the formation of cracks.

Five chemical analyses (Table 3) of uraninite were carried out using a LEO440 electron

microscope equipped with a Si-Li detector (EDS mode, 20 kV, 1.5 nA and 100 s counting time). The empirical formula, based on 2 O anions is  $(\text{U}_{0.65}^{4+}\text{Th}_{0.25}\text{Pb}_{0.21})_{\Sigma 1.11}\text{O}_2$ . However, the apparent cation excess suggests that the mean cation valence is actually higher, due to part of the uranium being present as  $\text{U}^{6+}$ . A formula with both  $\text{U}^{4+}$  and  $\text{U}^{6+}$  would be  $(\text{U}_{0.40}^{4+}\text{Th}_{0.22}\text{U}_{0.19}^{6+}\text{Pb}_{0.19})_{\Sigma 1.00}\text{O}_2$ .

## Crystallography

Powder X-ray diffraction data (for  $\text{CuK}\alpha$ ) were obtained using a Siemens D5000 diffractometer (Table 4). Unit-cell parameters refined from powder data are  $a = 10.575(2) \text{ \AA}$ ,  $V = 1182.6(8) \text{ \AA}^3$  and  $Z = 8$ . Calculated powder X-ray diffraction data for kenoplumbomicrolite are also given in Table 4. The crystallographic information files have been deposited with the Principal Editor of *Mineralogical Magazine* and are available as Supplementary material (see below).

Single-crystal X-ray studies (Bindi *et al.*, 2006) were carried out using a Bruker P4 automated diffractometer with graphite-monochromated  $\text{MoK}\alpha$  ( $\lambda = 0.71073 \text{ \AA}$ ) radiation. The unit-cell values are  $a = 10.571(1) \text{ \AA}$ ,  $V = 1181.3(2) \text{ \AA}^3$  and  $Z = 8$ . After the data reduction of the intensity dataset, the structure was refined in the space group  $Fd\bar{3}m$  to  $R_{\text{obs}} = 4.89\%$  and  $R_{\text{all}} = 5.79\%$ . The single-crystal X-ray study showed  $\text{Pb}^{2+}$  to be completely ordered at the *A* site. Because the *Y* site was only partially occupied, the  $\text{Pb}^{2+}$  may locally result as six-, seven-, or eight-fold coordinated. Whenever two neighbour *Y* sites are vacant,

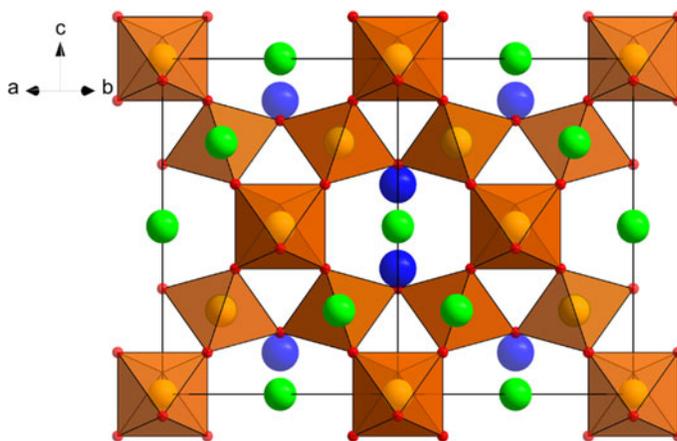


FIG. 4. Crystal structure of kenoplumbomicrolite down [110]. The orange polyhedra represent the *B*-site and the green, red and blue spheres represent *A*-, *X*- and *Y*-sites, respectively.

six  $X$  atoms around the  $A$  position form a trigonally flattened octahedron, while a trigonal scalenohedron occurs when two  $Y$  sites around  $A$  are occupied. When only one neighbour  $Y$  site is occupied, the  $A$  cation assumes an asymmetrical seven-fold pyramidal coordination. Due to the stereoactive electron lone pair of  $Pb^{2+}$ , this last coordination environment should be strongly preferred. The occupancy of the  $Y$  position was refined and the resulting electron density of 3.9(2) electrons agrees well with the occupancy of  $Y$  in the empirical formula,  $[\square_{0.52}(\text{OH})_{0.25}\text{O}_{0.23}]$ , corresponding to 4.09 electrons (Fig. 4). Experimental details and refined structural parameters used for the approved species in Tables 1 and 2, respectively are those reported in Bindi *et al.*, 2006.

## Conclusion

On the basis of the Ta dominance at the  $B$  site, the mineral from Ploskaya Mountain commonly known as ‘plumbomicrolite’ and previously described (Bindi *et al.*, 2006) maintains the root-name ‘microlite’; owing to the dominance of Pb at the  $A$  site the prefix ‘plumbo’ is also maintained. However, according to the IMA approved rules for nomenclature (Atencio *et al.*, 2010), the name ‘plumbomicrolite’ must be preceded by the prefix ‘keno’, which indicates that the  $Y$  site is dominantly vacant.

## Acknowledgements

We acknowledge the Brazilian agencies FAPESP (process 2014/50819-9 and 2013/03487-8), and all members of the IMA Commission on New Minerals, Nomenclature and Classification for their helpful suggestions and comments. We also thank Peter Williams for editorial handling and Stuart Mills and an anonymous reviewer for their detailed reviews.

## Supplementary material

To view supplementary material for this article, please visit <https://doi.org/10.1180/minmag.2017.081.082>

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