Osumilite-(Mg): Validation as a Mineral Species and New Data[1]

N. V. Chukanov[a, 2], I. V. Pekov[b], R. K. Rastsvetaeva[c], S. M. Aksenov[c], D. I. Belakovskiy[d], K. V. Van[e], W. Schüller[f], and B. Ternes[g]

[a] *Institute of Problems of Chemical Physics, Russian Academy of Sciences, Chernogolovka, Moscow oblast, 142432 Russia*
[b] *Faculty of Geology, Moscow State University, 119991 Moscow, Russia*
[c] *Institute of Crystallography, Russian Academy of Sciences, Leninskii pr. 59, Moscow, 117333, Russia*
[d] *Fersman Mineralogical Museum, Russian Academy of Sciences, Leninskii pr. 18-2, Moscow, 117071 Russia*
[e] *Institute of Experimental Mineralogy, Russian Academy of Sciences, Chernogolovka, Moscow oblast, 142432 Russia*
[f] *Straussenpesch 22, 53518 Adenau, Germany*
[g] *Bahnhofstrasse 45, 56727 Mayen, Germany*
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Abstract—Osumilite-(Mg), the Mg-dominant analogue of osumilite, has been approved by the CNMNC IMA as a new mineral species. The holotype sample has been found at Bellerberg, Eifel volcanic area, Germany. Fluorophlogopite, sanidine, cordierite, mullite, sillimanite, topaz, pseudobrookite and hematite are associated minerals. Osumilite-(Mg) occurs as short prismatic or thick tabular hexagonal crystals reaching 0.5×1 mm in size in the cavities in basaltic volcanic glasses at their contact with thermally metamorphosed xenoliths of pelitic rocks. The mineral is brittle, with Mohs' hardness 6.5. Cleavage was not observed. Color is blue to brown. $D_{meas} = 2.59(1)$, $D_{calc} = 2.595$ g/cm^3. No bands corresponding to H_2O and OH-groups are in the IR spectrum. Osumilite-(Mg) is uniaxial (+), $\omega = 1.539(2)$, $\varepsilon = 1.547(2)$. The chemical composition (electron microprobe, average of 5 point analyses, wt %) is: 0.08 Na_2O, 3.41 K_2O, 0.04 CaO, 7.98 MgO, 0.28 MnO, 21.57 Al_2O_3, 3.59 Fe_2O_3, 62.33 SiO_2, total 99.28. The empirical formula is: $(K_{0.72}Na_{0.03}Ca_{0.01})(Mg_{1.97}Mn_{0.04})[Al_{4.21}Fe^{3+}_{0.45}Si_{10.32}]O_{30}$. The simplified formula is: $KMg_2Al_3(Al_2Si_{10})O_{10}$. The crystal structure was refined on a single crystal, $R = 0.0294$. Osumilite-(Mg) is hexagonal, space group $P6/mcc$; $a = 10.0959(1)$, $c = 14.3282(2)$ Å, $V = 1264.79(6)$ Å^3, $Z = 2$. The strongest reflections in the X-ray powder diffraction pattern [d, Å (I %) (hkl)] are: 7.21 (37) (002), 5.064 (85) (110), 4.137 (45) (112), 3.736 (43) (202), 3.234 (100) (211), 2.932 (42) (114), 2.767 (51) (204). A type specimen is deposited in the Fersman Mineralogical Museum, Russian Academy of Sciences, Moscow, registration number 4174/1.

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INTRODUCTION

Osumilite, $(K,Na)(Fe^{2+},Mg)_2(Al,Fe^{3+})_3(Si_{10}Al_2)O_{30}$, is one of the most abundant minerals of the milarite group (Back, Mandarino, 2008) usually found in volcanic rocks, related metasomatic rocks (including contact-metamorphosed xenoliths of sedimentary rocks in lava), and metamorphic granulite complexes.

The milarite-group members are frequently attributed to ring silicates. In our opinion, this statement is not quite adequate. Their rating as tectosilicates is apparently more correct, because the structure of these minerals is based on the tetrahedral framework $M_3(Si,Al)_{12}O_{30}$ (M = Al, Fe, Be, B, Li, Zn, Mg), where double (double-floor) hexagonal rings $(Si,Al)_{12}O_{30}$ are linked by tetrahedra MO_4 via common apices. On the

official site of the Commission on New Minerals, Nomenclature, and Mineral Classification of the International Mineralogical Association (CNMNMC IMA), http:/pubsites.uws.edu.au/ima-cmnc/, the formula of osumilite is written as $KFe_2(Al_5Si_{10})O_{30}$, where tetrahedrally-coordinated framework cations are combined in parentheses. The Fe^{2+}-dominated site in osumilite is octahedrally coordinated. The large cations (K^+ together with Na^+ and Ca^{2+}) occupy the site between neighboring double rings.

The Mg-dominated (Mg > Fe at octahedral site) analog of osumilite has been described many times (Chinner and Dixon, 1973; Berg and Wheeler, 1976; Grew, 1982; Armbruster and Oberhlänsli, 1988; Balassone et al., 2008; Seryotkin et al., 2008); however, according to the aforementioned website, the mineral so far had status N (published without approval by CNMNC IMA). In the list of mineral species given on this site, for osumilite-(Mg) is given only one reference (Seryotkin et al., 2008), related to a technogenetic sample (from a burned coal dump) with the for-
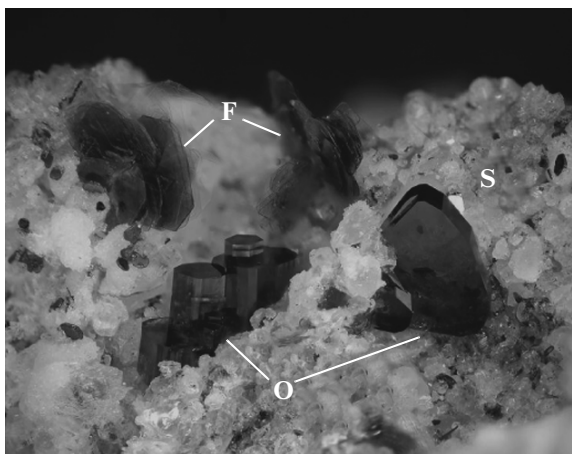

Fig. 1. Crystals of osumilite-(Mg) (O) with fluorophologopite (F) and sanidine (S). Width of image is 4 mm. Photo by M. Reinhardt.

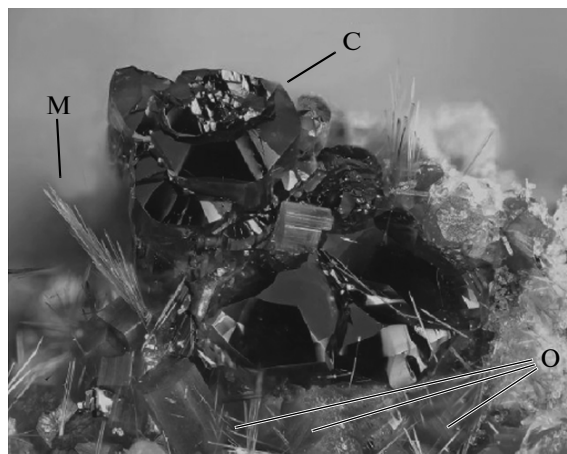**Fig. 2.** Crystals of osumilite-(Mg) (O) with cordierite (C) and mullite (M). Width of image is 2.5 mm. Photo by M. Reinhardt.

mula $KMg_2[Al(Fe^{2+})_2Si_{12}]O_{30}$, which is not consistent with the crystallochemical formula $K^C_{0.83} Na^{B'}_{0.10} (Mg_{1.78}Fe_{0.16}Mn_{0.03}Ti_{0.03})^A(Al_{2.88}Fe_{0.12})^{T2} (Al_{1.91} Si_{10.09})^{T1}O_{30}$ given by Seryotkin et al. (2008).

"Osumilite-(K,Mg)" $K(Mg,Fe)_2(Al,Fe)_3(Si, Al)_{12}O_{30}$ (Chinner and Dixon, 1973; *International* ..., 1980), has been discredited by CNMNC IMA (see site http:/pubsites.uws.edu.au/ ima-cnmnc/).

These contradictions and the indefinite status of osumilite-(Mg) impelled us to carry out its comprehensive study, including a determination of the chemical composition, physical properties, and crystal structure and to submit the results to CNMNC in order to strengthen the status of this mineral as an individual mineral species.

The holotype specimen of osumilite-(Mg) is deposited in the Fersman Mineralogical Museum, Russian Academy of Sciences, Moscow, registration number 4174/1.

OCCURRENCE, MORPHOLOGY, AND PHYSICAL PROPERTIES

The specimens with osumilite-(Mg) were collected in the operating Caspar basaltic quarry at Mount Bellerberg, near the town of Mayen, in the Eifel volcanic area of Rhineland−Palatinate (Rheinland−Pfalz), Germany. This mineral occurs as light blue to nearly colorless and brown short prismatic or thick tabular hexagonal crystals up to 0.5×0.8 mm in size, with clusters of the crystals reaching 1.5 mm across growing on the walls of cavities (bubbles) in the basaltic glass, close to the contact with the metamorphosed xenolith of clay rock, which is considered the major source of Al (Figs. 1, 2). Fluorophlogopite, sanidine, cordierite, mullite, sillimanite, topaz, pseudobrookite, and hematite are associated minerals.

Osumilite-(Mg) crystals are most frequently formed by the faces of prism $\{1.1.\overline{10}\}$ and pinacoid $\{0001\}$; the faces $\{1.0.\overline{11}\}$ and $\{1.1.\overline{20}\}$ are less frequent.

The luster of the mineral is vitreous; the streak is white. The Mohs' hardness is 6.5. The mineral is brittle, with conchoidal fractures. No cleavage has been observed. The density measured by the floatation method is 2.59(1) cm^3. The calculated density is 2.592 g/cm^3.

The IR spectrum of osumilite-(Mg) is close to that of osumilite (Fig. 3). The position (cm^{-1}) and assignment of the bands in the IR spectrum are as follows (s is strong band, sh is shoulder): 1138s, 1000sh, 950s (stretching vibrations of tetrahedra $(Si,Al)O_4$), 770sh, 745, 717, 652, 541s (vibrations of tetrahedral rings), 475, 440, 401s, 378s (combination modes of Si−O−Si bending vibrations and stretching vibrations of MgO_6 octahedra). In the IR spectrum of osumilite, the last band is shifted to 367 cm^{-1}, because Fe^{2+} is predominant over Mg at the octahedral site. The bands with the wave numbers higher than 1150 cm^{-1} are absent in the IR spectrum, testifying to the absence of H-, B- and C-bearing groups in the mineral.

Osumilite-(Mg) is optically uniaxial, positive, $\omega = 1.539(2)$, $\varepsilon = 1.547(2)$. The mineral is weakly pleochroic: colorless along E and pale blue along O.

CHEMICAL COMPOSITION

The chemical composition of osumilite-(Mg) was determined on a Tescan Vega II XMU SEM equipped with an INCAx-sight EDS that operates on tungsten cathode at an accelerating voltage of 15.7 kV. The current of the absorbed electrons on Co was 0.5 nA. The selection angle of X-ray radiation was 35°, and the focal distance between sample and detector was 25 mm.
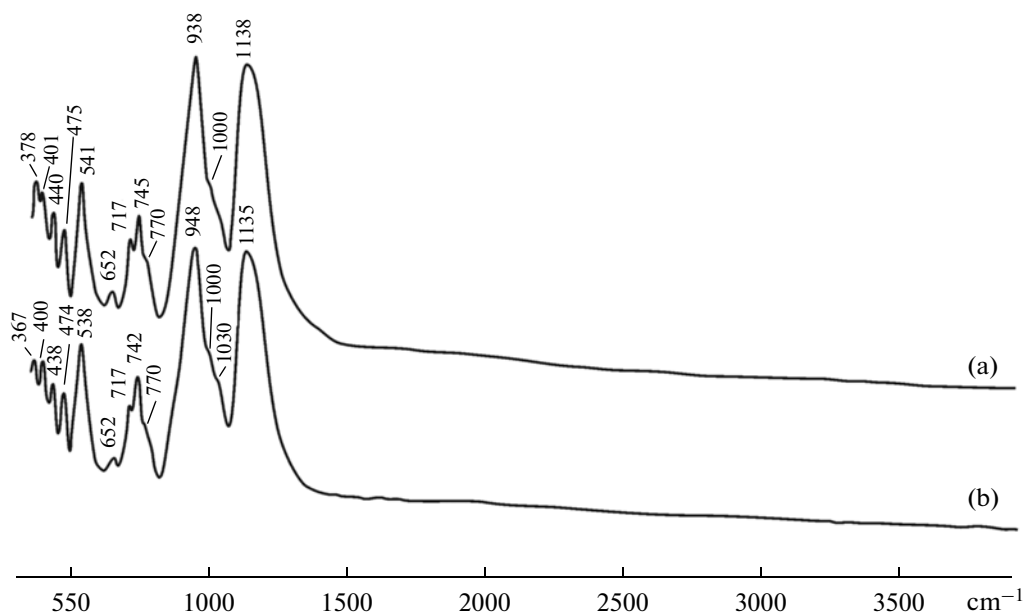
Fig. 3. IR spectra of (a) osumilite-(Mg) from Bellerberg and (b) osumilite with empirical formula $(K_{0.7}Na_{0.3})(Fe_{1.1}Mg_{0.8}Mn_{0.1})(Al_{2.5}Fe_{0.5})(Al_{1.7}Si_{10.3})O_{30}$ from Sakkabira Hill, near Sakurajima volcano, Osumi Peninsula, Kyushu, Japan.

Owing to the absence of characteristic bands assigned to H-, B-, and C-bearing groups in the IR spectrum of osumilite-(Mg), H_2O, B_2O_3, and CO_2 were not determined.

The electron microprobe data on the mineral are given in Table 1.

The empirical formula of osumilite-(Mg), calculated on the basis of 30 O atoms in the assumption that all Fe in the studied sample is Fe^{3+}, is as follows: $(K_{0.72}Na_{0.03}Ca_{0.01})(Mg_{1.97}Mn_{0.04})[Al_{4.21}Fe_{0.45}Si_{10.32}]O_{30}$. The total content of Mg + Mn + Al + Fe + Si in such a mode of formula calculation is 16.99, which is consistent with the assumption of the trivalent state of iron. A formula calculated with Fe^{2+} on the basis of 30 O atoms might have a significant deficiency of cations that contradicts the structural data, including the refined number of electrons at the structural sites. In addition, the trivalent state of iron is supported by the nearly ideal compatibility of the physical properties and the chemical composition calculated from the Gladstone–Dale equation $(1 - Kp/Kc) = -0.001$ (*superior*). Fe^{3+} ions are placed at the tetrahedral site, because, otherwise, the partial tetrahedral coordination of Mg would be inconsistent with the structural data, namely, with the number of electrons at sites and with interatomic distances (see below).

The holotype specimen of osumilite-(Mg) is pale blue, indicating a probable charge transfer between Fe^{2+} and Fe^{3+}. Taking this into account, a small amount of Fe^{2+} in the mineral cannot be excluded, although, according to the aforementioned reasons, this amount most likely does not exceed $0.0n$ apfu. To compare, the similar color of beryl variety aquama-

rine, which is structure-related to osumilite, is observed at the Fe^{2+} content of about $0.n$ wt %.

The idealized formula of osumilite-(Mg) derived from the structural data is $KMg_2[Al_3(Al_2Si_{10})O_{30}]$, where the cations of the double-floor ring are in parentheses and the tetrahedral framework is in brackets.

X-RAY CRYSTALLOGRAPHY AND CRYSTAL STRUCTURE

The X-ray powder diffraction pattern of osumilite-(Mg) (Table 2) was recorded with a Stoe IPDS II single-crystal diffractometer with an image plate detector, MoK_{α} radiation, an accelerating voltage of 45 kV, and a current of 30 mA. The experiment has been car-

Table 1. Chemical composition of osumilite-(Mg) (average of 5 point analyses)

Component	Wt %	Range
Na_2O	0.08	0–0.16
K_2O	3.41	3.24–3.60
CaO	0.04	0–0.09
MgO	7.98	7.85–8.11
MnO	0.28	0.22–0.36
Al_2O_3	21.57	21.19–21.93
Fe_2O_3	3.59	3.43–3.78
SiO_2	62.33	61.88–63.02
Total	99.28	

F, P, S, Ti, V, Cr, Ni, Zn, Y, Zr, REE, Th, and U were not detected.

Table 2. X-ray powder diffraction data of osumilite-(Mg)

I_{obs}, %	d_{obs}, Å	d_{calc}, Å	hkl	I_{obs}, %	d_{obs}, Å	d_{calc}, Å	hkl
37	7.21	7.145	002	21	1.851	1.853, 1.850	315, 412
36	5.538	5.541	102	7	1.793	1.786	008
85	5.064	5.067	110	17	1.738	1.739, 1.735	217, 226
26	4.393	4.389	200	8	1.684	1.685	118
45	4.137	4.133	112	5	1.652	1.654, 1.648	208, 421
43	3.736	3.739	202	2	1.587	1.591	415
22	3.587	3.573	004	4	1.562	1.564	317
100	3.234	3.232	211	7	1.528	1.527, 1.525	334, 308
42	2.932	2.920	114	1	1.492	1.493	416
51	2.767	2.771	204	5	1.458	1.460	228
16	2.522	2.534	220	9	1.431	1.432, 1.429	219, 0.0.10
11	2.398	2.400	311	7	1.395	1.397	417
2	2.292	2.298	106	3	1.373	1.375	1.1.10
9	2.162	2.165, 2.156	215, 116	3	1.354	1.354	604
4	2.086	2.093	206	5	1.333	1.336, 1.330	328, 319
15	2.007	2.014, 2.012, 1.994	320, 314, 321	2	1.312	1.315, 1.312	516, 2.1.10
11	1.893	1.898	411	3	1.306	1.308, 1.306	524, 418

ried out using the Gandolfi method with revolution on two axes. The distance between sample and detector was 200 mm; the measurement time was 40 min.

All reflections are well indexed in the hexagonal unit cell, with dimensions refined on the basis of the X-ray powder diffraction data: $a = 10.135(7)$, $c = 14.29(2)$ Å, $V = 1271(3)$ Å^3.

The single-crystal X-ray diffraction data were collected with an Agilent SuperNova CCD diffractome-

Fig. 4. Crystal structure of osumilite-(Mg) projected onto *ab* plane.

ter using MoK_α radiation. The calculated hexagonal unit-cell dimensions (space group $P6/mcc$) are: $a = 10.0959(1)$, $c = 14.3282(2)$ Å, $V = 1264.79(6)$ Å^3, $Z = 2$. The crystal structure of osumilite-(Mg) was solved by the charge flipping method and refined with the Jana2006 program (Petricek et al., 2006) in anisotropic approximation using 1065 unique reflections with $F > 3\sigma F$. The R-factor of refinement is 2.94%.

The crystal structure of osumilite-(Mg) from Bellerberg is identical to that of other osumilite-(Mg) and osumilite samples (Miyashiro, 1956; Brown and Grens, 1969; Armbruster and Oberhänsli, 1988; Balassone et al., 2008; Seryotkin et al., 2008).

Comparison of the chemical composition of the mineral, interatomic distances, refined number of electrons, and bond valence sums for the sites of Mg, Al, and Si results in the following arrangement of constituents in the sample studied here:

—octahedral site of Mg is occupied only by Mg;

—tetrahedral site of Al is occupied by Al with minor Fe^{3+};

—tetrahedral site of Si is occupied by Si with minor Al.

The distance Al–O (1.7768 Å) is consistent with Fe^{3+} at the site of Al, rather than the larger cations Fe^{2+} or Mg^{2+}.
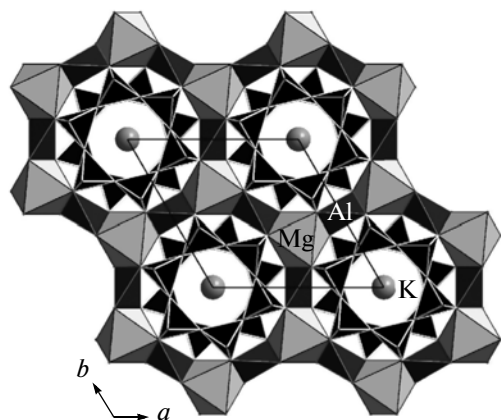
Table 3. Major crystallographic parameters of osumilite and data of the monocrystal experiment

Chemical formula	$K_{0.7}Mg_2(Al_{2.5}Fe_{0.5})(Si_{10.3}Al_{1.7})O_{30}$
Space group, Z	Hexagonal, $P6/mcc$, 2
a, Å	10.0959(1)
c, Å	14.3282(2)
γ, degree	120
V, Å^3	1264.79(6)
D_x, g/cm^3	2.59
Radiation, λ, Å	Mo_{K_α}, 0.71073
μ, mm^{-1}	1.256
T, K	293
Size of crystal	$0.25 \times 0.21 \times 0.15$
Diffractometer	Agilent SuperNova, CCD-detector
Scanning type	ω-scanning
θ_{max}, degree	37.72
Ranges of h, k, and l	$\overline{16} < h < 17$; $\overline{16} < k < 17$; $\overline{24} < l < 24$
Number of observed reflections	23619
Number of unique reflections (N_1), R_{int}	1172, 2.84
Unique reflections $I > 3\sigma(I)$ (N_2)	1065
Method of refinement	Least square method by F
Number of refined parameters	41
Weight scheme	$1/(\sigma^2(F) + 0.001225F^2)$
Uncertainty factor	
R_1 by N_2	2.94
wR_2 by N_1	4.72
S	1.17
$\Delta\rho_{max}/\Delta\rho_{min}$, e/Å^3	0.56/−0.67
Program	Jana 2006

The site of K occurs in the channels of the framework. Refinement of the structure under the assumption of its complete occupancy led to unrealistic isotropic displacement parameter $U_{iso} = 0.043$, which may indicate a weak splitting of this site.

Refinement at fixed parameter $U_{iso} = 0.02$ provided a degree of its occupancy 0.692(5) that is consistent with the electron microprobe data indicating a deficiency of K.

Major features of the composition and crystal structure of the mineral are reflected in its crystallochemical formula ($Z = 2$): $[K_{0.7}]^{XII}$ $[Mg_2]^{VI}$

Table 4. Site coordinates in the structure of osumilite-(Mg) and refined number of electrons at the cation sites

Element	x/a	y/b	z/c	e_{ref}
K	0	0	0.25	13.3
Mg	0.3333	0.6667	0.25	12.0
Al	0.5	0	0.25	15.2
Si	0.1059(1)	0.3533(3)	0.1077(1)	13.9
O1	0.1226(1)	0.4073(1)	0	
O2	0.2173(1)	0.2850(1)	0.1309(1)	
O3	0.1407(1)	0.4936(1)	0.1788(1)	

Table 5. Anisotropic displacement parameters (Å^2) for osumilite-(Mg)

Element	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
K	0.0299(3)	0.0299(3)	0.0352(5)	0.0150(2)	0	0
Mg	0.0048(2)	0.0048(2)	0.0072(2)	0.0024(1)	0	0
Al	0.0185(2)	0.0083(2)	0.0116(2)	0.0042(1)	0	0
Si	0.0082(1)	0.0104(1)	0.0077(1)	0.0050(1)	−0.0001(1)	−0.0014(1)
O1	0.0267(4)	0.0209(4)	0.0099(3)	0.0113(3)	0	0
O2	0.0248(3)	0.0317(4)	0.0257(3)	0.0226(3)	0.0005(2)	−0.0019(3)
O3	0.0152(2)	0.0158(2)	0.0133(2)	0.0090(2)	−0.0030(2)	−0.0049(2)

Table 6. Parameters of coordination polyhedra in the structure of osumilite-(Mg)

Element	Distance, Å
K	K–O2 = 3.1128 × 12
Mg	Mg–O3 = 2.1163 × 6
Al	Al–O3 = 1.7768 × 4
Si	Si–O1 = 1.6178
	Si–O2 = 1.6200
	Si–O2' = 1.6313
	Si–O3 = 1.6340

$[Al_{2.5}Fe_{0.5}]^{IV} [Si_{10.3}Al_{1.7}]^{IV} O_{30}$, where the compositions of cation sites are in brackets, while their coordination numbers are shown by Roman numerals.

DISCUSSION

Osumilite was found for the first time in material collected near the volcano Sakuradzima, Osumi Peninsula, Kyusyu, Japan. In the structure of this mineral, the octahedral sites are predominantly occupied by Fe^{2+} (Miyashiro, 1956). A specimen of osumilite from the location of its original discovery was studied by us, and its empirical formula is as follows: $(K_{0.7}Na_{0.3})(Fe_{1.1}Mg_{0.8}Mn_{0.1})(Al_{2.5}Fe_{0.5})(Al_{1.7}Si_{10.3})O_{30}$.

Previously, Armbruster and Oberhänsli (1988) studied the crystal structures of the osumilite–osumilite-(Mg) solid solution series with a widely variable Fe : Mg value at the octahedral site. It is noteworthy that, among the specimens studied in the cited paper, the Mg-poorest specimen of osumulite (Mg 0.11 apfu, Fe : (Mg + Fe) = 0.94) came from the contact metamorphosed xenolith at Bellerberg.

Later, the crystal structure of Fe^{2+}-dominant osumilite was examined in the sample from the Arci volcanic massif in Sardinia (Elmi et al., 2010). The crystallochemical formula of that sample is $(K_{0.729}Na_{0.029})(Mg_{0.735}Mn_{0.091}Fe_{1.184})(Al_{2.706}Fe_{0.294})(Si_{10.498}Al_{1.502})O_{30}$.

Thus, the existence of natural osumilite as an Fe^{2+}-dominant (at the octahedral site) mineral is undoubted. However, it should be noted that in the list of minerals given on the website of CNMNC IMA, osumilite is mistakenly referenced as Balassone et al. (2008), who reported osumilite-(Mg) with an Fe content of 0.247 apfu.

The crystal structure of osumilite with Mg : Fe^{2+} = 1 : 1 at the octahedral site was previously studied by Brown and Gibbs (1969). Armbruster and Oberhänsli (1988), Balassone et al. (2008), and Seryotkin et al. (2008) reported the structure of osumilite-(Mg). The complete solid solution between osumilite and osumilite-(Mg) with the variable Mg : Fe value at the octahedral site is evident.

It should be noted that the Mg content at the octahedral site in osumilite from the Bellerberg xenoliths ranges from 5.5 to 98 at % (Armbruster and Oberhänsli, 1988). This indicates a substantial role of local concentrations of Fe and Mg in osumilite crystallized as a result of contact metamorphism of xenoliths and, in particular, the role of the chemical composition of the xenoliths themselves. The oxidative environment may facilitate the formation of Mg-rich varieties of osumilite; Fe becomes trivalent and concentrates at tetrahedra rather than at octahedra, substituting Al (as in the sample studied here), and also entering into associated hematite, pseudobrookite, and other minerals containing Fe^{3+}.

Schreyer et al. (1983) has shown that osumilites crystallized at low pressure are characterized by a low content of large cations (K + Na + Ca). This is supported by the members of the osumilite–osumilite-(Mg) solid solution series from the Bellerberg xenoliths, notable for a significant cation deficiency of the K-site reaching 62% (Armbruster and Oberhänsli, 1988)

Table 7. Comparative data for osumilite-(Mg) and osumilite

Parameter	Osumilite-(Mg)	Osumilite
Formula	$KMg_2Al_3(Al_2Si_{10})O_{30}$	$KFe_2^{2+}Al_3(Al_2Si_{10})O_{30}$
Symmetry	Hexagonal, $P6/mcc$	Hexagonal, $P6/mcc$
a, Å	10.0959	10.08−10.17
c, Å	14.3282	14.26−14.35
Z	2	2
Strong reflections in X-ray powder diffraction pattern, d, Å, (I)	7.21 (37)	7.17 (strong)
	5.064 (85)	5.08 (strong)
	4.137 (45)	4.41 (medium)
	3.736 (43)	3.74 (medium)
	3.324 (100)	3.24 (very strong)
	2.932 (42)	2.930 (strong)
	2.767 (51)	2.776 (medium)
Optical parameters		
ω	1.539	1.539−1.547
ε	1.547	1.545−1.551
Optical sign	(+)	(+) or (−)
Density, g/cm^3	2.59 (meas)	2.58−2.68 (meas)
	2.592 (calc)	2.71 (calc)
Reference	This study	Deer et al., 1986; Miyashiro, 1956; Armbruster, Oberhansli, 1988

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