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Crystal structure of $K_3EuSi_2O_7$

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Abstract: As part of research on the flux technique for growing alkali rare-earth elements (REE) containing silicates, tripotassium europium disilicate, $K_3EuSi_2O_7$, was synthesized and characterized by single-crystal X-ray diffraction. It crystallizes in the space group $P6_3/mcm$. In the crystal structure of the title compound, one part of the Eu cations are in a slightly distorted octahedral coordination and the other part are in an ideal trigonal prismatic coordination environment. The disilicate Si_2O_7 groups connect four EuO_6 octahedra and one EuO_6 trigonal prism. Three differently coordinated potassium cations are located between them. Silicates containing the larger rare earth elements usually crystallize in a structure that contains the rare-earth cation in both a slightly distorted octahedral and an ideal trigonal prismatic coordination environment.

Keywords: alkali rare-earth silicates; crystal structure; flux synthesis; single-crystal X-ray diffraction.

INTRODUCTION

Silicates containing rare-earth elements (REE silicates) have been the subject of intense research in recent decades. Many studies were performed on REE silicates because of their wide range of optical and magnetic properties combined with high thermal stability,[1–9] which make them very interesting and promising materials for different applications. REE silicates also exhibit broad structural diversity.[10] A highly adaptive framework can incorporate almost all REEs and this opens the possibility of synthesizing numerous structures and structural series of REE-containing silicates. Small but systematic variations in the cation radii among the lanthanides allow extensive cation substitutions.

Several ternary potassium–REE silicates with the stoichiometric composition $K_3REESi_2O_7$ that crystallize in the space groups $P6_3/mcm$ and $P6_3/mmc$ have hitherto been described: $K_3EuSi_2O_7$,[11] $K_3NdSi_2O_7$,[12] $K_3\mathit{REE}Si_2O_7$ (REE = Gd–

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–Lu),[13] $K_3ScSi_2O_7$,[14] $K_3YSi_2O_7$ (2 polymorphs),[6] $K_3SmSi_2O_7$[7] and $K_3ErSi_2O_7$ (2 polymorphs).[15]

During our investigations focused on the synthesis of new potassium-REE silicates, single crystals of $K_3EuSi_2O_7$ were obtained. $K_3EuSi_2O_7$ crystallizes in the hexagonal space group *P*6$_3$/*mcm* (No. 193) and is isostructural with $K_3NdSi_2O_7$,[12] $K_3\mathit{REE}Si_2O_7$ (REE = Gd–Yb),[13] $K_3YSi_2O_7$,[6] $K_3SmSi_2O_7$[7] and $K_3ErSi_2O_7$.[15] The reported compound was already known in the literature. Bondar and co-authors[11] stated the existence of this phase, however, they reported only the unit cell parameters (a = 9.98(1), c = 14.44(2) Å), space group (*P*6$_3$/*mcm*), chemical composition and described the morphology of crystals. Myers[16] outlined the successful synthesis of $K_3\mathit{REE}Si_2O_7$ (REE = Eu, Y, Sc), but without any peer-reviewed data for $K_3EuSi_2O_7$. Thus, the crystal structure of this compound, $K_3EuSi_2O_7$, has not hitherto been described in the literature.

EXPERIMENTAL

Materials and measurements

All the materials were of analytical reagent grade and used as received without further purification. The crystal structure of $K_3EuSi_2O_7$ was analyzed by the single-crystal X-ray diffraction method. Diffraction experiments were conducted on a Gemini S (Oxford Diffraction) four-circle diffractometer equipped with a Mo-anode sealed tube and a Sapphire3 CCD detector. Diffraction data were processed with CrysAlis$^{\mathrm{Pro}}$.[17] The synthesized products were tested using X-ray powder diffraction (XRPD). A small amount of material was finely ground and tested on a Philips PW1710 X-ray powder diffractometer at room temperature using Bragg––Brentano geometry and CuKα radiation. The diffractometer was operated at 40 kV and 30 mA, while the 2θ scan range was from 3 to 60°, with a step size of 0.02° and time per step of 1.25 s. PDXL 2 software[18] was used for analysis of the powder diffraction data. Scanning electron microscopy (SEM) was used for the morphological characterization. The analyses were performed on a single crystal using a JEOL JSM-6610LV instrument equipped with an Oxford INCA Energy 350 EDS detector.

Synthesis and crystallization

The following chemicals were used in the high-temperature flux synthesis experiment: Eu_2O_3 (Sigma–Aldrich, 99.99 %), SiO_2 (Merck, p.a.) and KF (Centrohem, 99 %). A total of 0.055 g of a mixture of Eu_2O_3 and SiO_2 in a molar ratio of 1:4 was added to 1 g of KF. The resulting mixture was homogenized in an agate mortar, transferred to a platinum crucible covered with a platinum lid and placed in a resistance-heated furnace. The mixture was heated to 1173 K at a rate of 200 K h^{-1} and kept for 10 h at that temperature. The temperature was lowered to 973 K at a rate of 2 K h^{-1} and then the furnace was switched off. After cooling to room temperature, the resulting material was manually removed from the crucible, washed with distilled water and dried in air. Bow-tie aggregate colorless crystals up to 300 μm in size embedded in a polycrystalline matrix were observed.

X-Ray structure determination

A selected elongated prismatic colorless single crystal was fixed on glass fiber using nail polish as glue and X-ray diffraction data were collected at room-temperature. Relevant information on the crystal data, data collection and structure refinement are compiled in Table I.

An analytical absorption correction based on indexed crystal faces using the procedure of Clark & Reid[19] in combination with an empirical absorption correction using spherical harmonics (implemented in SCALE3 ABSPACK scaling algorithm) was applied during the data reduction. The $K_3EuSi_2O_7$ crystallizes in the space group $P6_3/mcm$. Since it is isostructural with $K_3ErSi_2O_7$,[15] atomic coordinates of this structural analogue were used as a starting model for structure refinement. When Er^{3+} was replaced with Eu^{3+}, the model converged rapidly, and the crystal structure was refined to $R = 3.5$ %. The structure was refined on F^2 by full-matrix least-squares techniques using SHELXL programs.[20] Selected bond lengths and angles are presented in Table II. All figures of the crystal structures were prepared using the Vesta program.[21]

TABLE I. Experimental details

Crystal data		Data collection	
Chemical formula	$K_3EuSi_2O_7$	Diffractometer	Gemini S (Oxford Diffraction)
M_r	437.44	Absorption correction	Analytical + empirical
Crystal system	Hexagonal	Measured reflections	10007
Space group	$P6_3/mcm$	Independent reflections	528
Temperature, K	298	Observed ($I > 2\sigma(I)$) reflections	392
a / Å	9.9512 (3)	R_{int}	0.077
c / Å	14.4480 (4)	$(\sin \theta/\lambda)_{max}$ / Å^{-1}	0.641
α / °	90	Refinement	
γ / °	120	$R[F^2 > 2\sigma(F^2)]$	0.035
V / Å^3	1239.05 (8)	$wR(F^2)$	0.069
Z	6	S	1.13
Radiation type	MoKα	No. of reflections	528
μ / mm^{-1}	9.39	No. of parameters	40
Crystal size, mm	0.27×0.04×0.04	$\Delta\rho_{max}$, $\Delta\rho_{min}$ / e Å^{-3}	1.01, –0.69

TABLE II. Selected geometric parameters for $K_3EuSi_2O_7$. Symmetry codes: $^{i}x-y, -y, z$; $^{ii}-y+1, x-y, z$; $^{iii}-x+1, -x+y, z$; $^{iv}y, -x+y, -z$; $^{v}x, x-y, -z$; $^{v}-x+1, -x+y, -z+1/2$; $^{vii}x-y, -y, -z+1/2$; $^{viii}y, x-1, z$; $^{ix}y, x-1, -z+1/2$; $^{x}-x+y, y, -z$; $^{xi}y, x, z$; $^{xii}-y, -x, -z$; $^{xiii}-x, -y, -z$; $^{xiv}x-y, x-1, -z$; $^{xv}-x+1, -y, -z$; $^{xvi}y, x, -z+1/2$; $^{xvii}x, y, -z+1/2$; $^{(xviii}y+1, x, z$

Bond	Bond length, Å	Bond	Bond length, Å
K1–O1	2.770(5)	K3–O2^{xii}	2.754(7)
K1–O1^{i}	2.770(5)	K3–O2^{xiii}	2.754(7)
K1–O2^{i}	2.962(3)	K3–O2	2.754(7)
K1–O2	2.962(3)	Eu1–O1^{iv}	2.282(4)
K1–O1^{ii}	3.006(6)	Eu1–O1^{i}	2.282(4)
K1–O1^{iii}	3.006(6)	Eu1–O1^{xiv}	2.282(4)
K1–O1^{iv}	3.049(5)	Eu1–O1^{iii}	2.282(4)
K1–O1^{v}	3.049(5)	Eu1–O1^{viii}	2.282(4)
K2–O1^{i}	2.923(4)	Eu1–O1^{xv}	2.282(4)
K2–O1^{vi}	2.923(4)	Eu2–O2^{vii}	2.334(7)
K2–O1^{vii}	2.923(4)	Eu2–O2	2.334(7)
K2–O1^{iii}	2.923(4)	Eu2–O2^{xi}	2.334(7)
K2–O1^{viii}	2.923(4)	Eu2–O2^{i}	2.334(7)

TABLE II. Continued

Bond	Bond length, Å	Bond	Bond length, Å
K2–O1^{ix}	2.923(4)	Eu2–O2^{xvi}	2.334(7)
K2–O3^{viii}	3.029(3)	Eu2–O2^{xvii}	2.334(7)
K2–O3	3.029(3)	Si1–O1^{iii}	1.616(5)
K2–O3^{iii}	3.029(3)	Si1–O1^{ii}	1.616(5)
K3–O2^{i}	2.754(7)	Si1–O2^{xviii}	1.622(7)
K3–O2^{x}	2.754(7)	Si1–O3	1.661(4)
K3–O2^{xi}	2.754(7)		
Bond	**Bond angle, °**	**Bond**	**Bond angle, °**
O1^{iii}–Si1–O1^{ii}	111.0(4)	O1^{ii}–Si1–O3	106.7(2)
O1^{iii}–Si1–O2^{xviii}	110.9(2)	O2^{xviii}–Si1–O3	110.5(4)
O1^{ii}–Si1–O2^{xviii}	110.9(2)	Si1–O3–Si1^{xvii}	136.7(6)
O1^{iii}–Si1–O3	106.7(2)		

RESULTS AND DISCUSSION

Description of the crystal structure

As a result of an extensive study of the flux technique for growing new alkali rare-earth elements (REE) containing silicates, $K_3EuSi_2O_7$ was synthesized and characterized by single-crystal X-ray diffraction. The compound crystallizes in the space group $P6_3/mcm$ and is isostructural with disilicates $K_3ErSi_2O_7$[15] and $K_3YbSi_2O_7$.[22] The crystal structure is comparable to the sorosilicate structure type reported earlier by Vidican and co-authors.[13]

The crystal structure of the title compound consists of Si_2O_7 groups and EuO_6 polyhedra that form a 3D framework with potassium ions in the holes (Fig. 1).

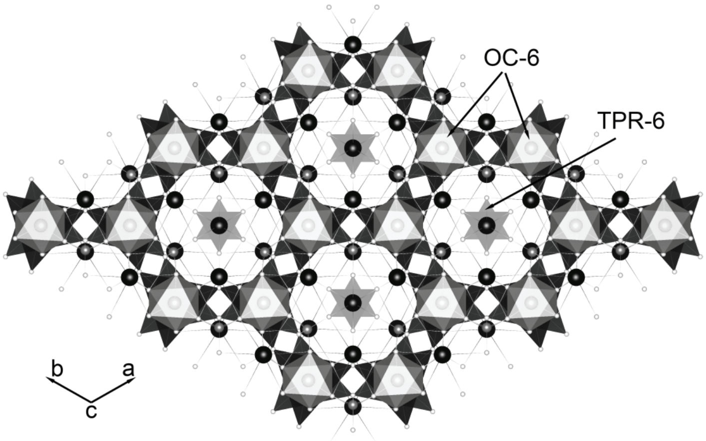

Fig. 1. The framework of the crystal structure of $K_3EuSi_2O_7$ along [001]. Color code: K atoms and polyhedra are black, EuO_6 polyhedra are grey (octahedra: OC-6; trigonal prisms: TPR-6), Si atoms are dark grey and O atoms are white.

The disilicate groups Si_2O_7 connect four EuO_6 octahedra (OC-6), as well as one EuO_6 trigonal prism (TPR-6, Fig. 2). Three differently coordinated potassium cations are located between them. The structure can also be described as regular alternation of two types of layers, which are parallel to the (001) plane: (I) octahedral layers and (II) sorosilicate layers, formed by a mixture of Si_2O_7 groups and K2 O_{6+3} polyhedra (Fig. 3).

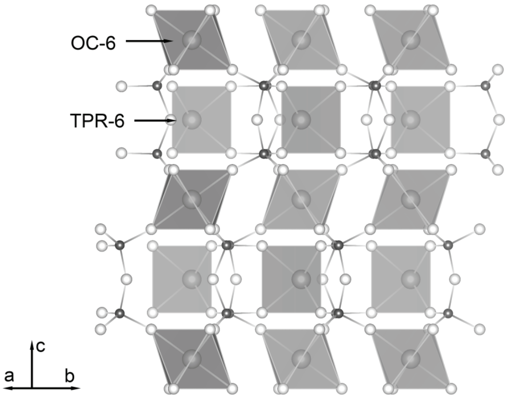

Fig. 2. The Si_2O_7 groups and EuO_6 polyhedra (octahedra: OC-6; trigonal prisms: TPR-6) in the crystal structure of $K_3EuSi_2O_7$ oriented approximately along [120] (*c*-axis is vertical) showing their connections. Potassium cations have been omitted for clarity. Color code: Eu atoms and polyhedra are grey, Si atoms are dark grey and O atoms are white.

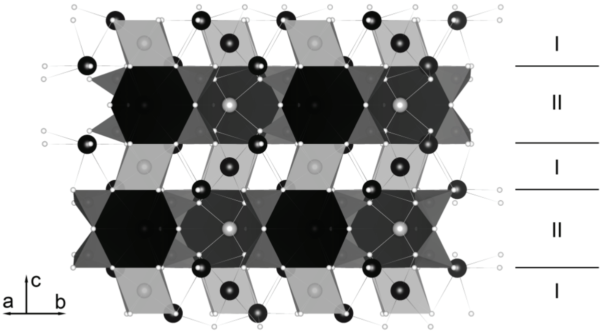

Fig. 3. Projection of structure along [120] showing two types of regularly alternating layers, which are parallel to the (001) plane: (I) octahedral layers and (II) sorosilicate layers. K atoms and polyhedra are black, Eu atoms and polyhedra are grey, SiO_4 polyhedra are dark grey and O atoms are white.

The structure is characterized by two crystallographically distinct six-coordinated sites occupied by Eu1 and Eu2: $Eu1O_6$ is an octahedron (OC-6) and $Eu2O_6$ is a trigonal prism (TPR-6). Two types of polyhedra around europium cations are distinguished by a small difference in the Eu–O distances: all Eu1–O distances are 2.282(4) Å, while all Eu2–O bond lengths are 2.334(7) Å. Each $Eu1O_6$ octahedron (OC-6) shares its six corners with six different Si_2O_7 groups, while each Er_2O_6 trigonal prism (TPR-6) also shares six corners, but only with three different Si_2O_7 groups.

Each Si_2O_7 group is connected to five different cations, *i.e.*, four Eu1 and one Eu2. The disilicate group consists of two SiO_4 tetrahedra sharing an O atom, denoted O3, with a bridging bond angle Si–O–Si of 136.7(6)°. The observed individual Si–O distances are within the acceptable range for silicate structures. The bridging Si–O bond (1.661(4) Å) is expectedly longer than the nonbridging Si–O bonds (1.616(5) and 1.622(7) Å). The O–Si–O bond angles range from 106.7(2) to 111.0(4)°.

The three potassium cations show three different coordination environments. The coordination sphere of K1 includes eight oxygen ions and forms a $K1O_{2+4+2}$ coordination polyhedron that could be described as a distorted hexagonal pyramid with a split apex (distances range from 2.770(5) to 3.049(5) Å). The K2 atom is coordinated by nine oxygen atoms with distances from 2.923(4) to 3.029(3) Å providing a regular tricapped trigonal prism ($K2O_{6+3}$). The coordination of the K3 atom (CN=6) could be described as an undistorted trigonal antiprism (K–O length = 2.754 (7) Å).

Bond-valence calculations showed that the bond valences are well balanced and the K–O, Eu–O and Si–O bond lengths are consistent with the presence of K^+, Eu^{3+}, Si^{4+} and O^{2-} in the structure. Only the bond-valence sum for Eu1 is oversaturated (3.42 v.u.), which could be attributed to the environment of the $Eu1O_6$ octahedron. All oxygen atoms from the $Eu1O_6$ octahedron are common to all adjacent polyhedra around the K1 and K2 atoms for which BVS shows slightly undersaturated values. This means that slightly deviating bonds are formed in order to satisfy the local valence disagreement due to structural disorder. These values are in accordance with the literature data for $K_3REESi_2O_7$[13] that contains the rare-earth cation in both slightly distorted octahedral and an ideal trigonal prismatic coordination environment. The results of bond-valence-sum calculations (VaList software;[23] bond valence parameters: Brown & Altermatt[24] and Brese & O'Keeffe[25]), are presented in Table III.

TABLE III. Bond valence sums for the cations and anions in $K_3EuSi_2O_7$

Atom	Bond valence, v. u.						Σv_{ij} / v. u.
	K1[a]	K2[a]	K3[a]	Eu1[a]	Eu2[a]	Si[b]	
O1	0.178×2 0.094×2 0.084×2	0.118×6		0.57×6		1.022×2	2.066
O2	0.106×2		0.186×6		0.495×6	1.005	1.898
O3		0.089×3				0.905	1.988
Σv_{ij} / v. u.	0.924	0.975	1.116	3.42	2.97	3.954	

[a]Brown & Altermatt[24]; [b]Brese & O'Keeffe[25]

X-Ray powder diffraction (XRPD)

Collected XRPD data are shown in Fig. 4. A diffraction pattern calculated using the CIF file obtained from the single-crystal data of $K_3EuSi_2O_7$ was overlaid. The experimental XRPD patterns are in good agreement with those calculated from the CIF.

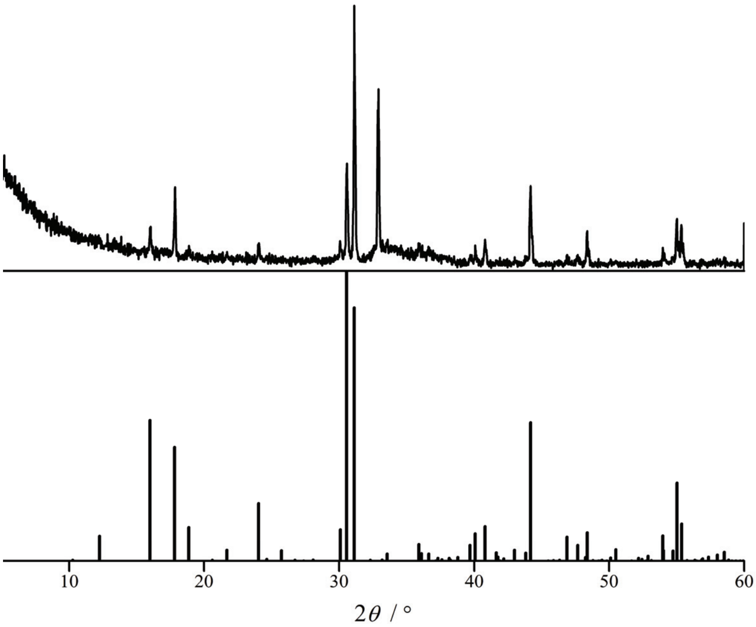

Fig. 4. X-ray powder diffraction pattern of $K_3EuSi_2O_7$. Upper pattern represents experimental data while lower pattern represents overlaid positions of Bragg's peaks calculated from $K_3EuSi_2O_7$ CIF.

Scanning electron microscopy (SEM)

The crystal morphology of a single $K_3EuSi_2O_7$ crystal obtained by SEM is shown in Fig. 5. An elongated prismatic crystal is clearly visible. The crystal

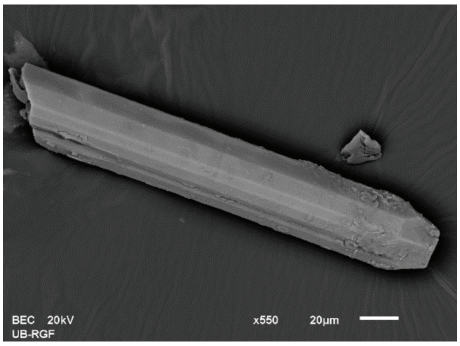

Fig. 5. A scanning electron microscope (SEM) picture of a $K_3EuSi_2O_7$ crystal.

appears to be homogeneous. Similar crystal morphology was also reported by Bondar and co-authors,[11] although their crystals were much thicker and less elongated.

CONCLUSIONS

Single crystals of $K_3EuSi_2O_7$ were prepared by high-temperature flux crystal growth and characterized by single-crystal X-ray diffraction, powder X-ray diffraction and SEM analysis. The compound crystallizes like disilicates containing larger REEs, *i.e.*, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb and Y, with the exception of Er and Y, which can crystallize as polymorphs in space groups $P6_3/mcm$ and $P6_3/mmc$. Their effective ionic radii ranged from 0.983 Å for $^{VI}Nd^{3+}$ to 0.868 Å for $^{VI}Yb^{3+}$ ($^{VI}Eu^{3+}$ = 0.947 Å).[26] The structure of these compounds contain the rare-earth cation in both a slightly distorted octahedral and an ideal trigonal prismatic coordination environment.

SUPPLEMENTARY MATERIAL

The crystallographic data for this paper have been deposited at the Cambridge Crystallographic Data Centre under deposition number CCDC 2062974.

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ИЗВОД

КРИСТАЛНА СТРУКТУРА $K_3EuSi_2O_7$

САБИНА З. КОВАЧ, ПРЕДРАГ З. ДАБИЋ и АЛЕКСАНДАР С. КРЕМЕНОВИЋ

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У оквиру испитивања алкалних силиката елемената ретких земаља добијених методом флукса из растопа, синтетисан је трикалијум европијум дисиликат, $K_3EuSi_2O_7$. Кристална структура одређена је методом рендгенске дифракције на монокристалу. Кристалише у просторној групи $P6_3/mcm$. У кристалној структури испитиваног једињења, део Eu катјона је у благо деформисаној октаедарској координацији, а други део је у идеалном тригонално-призматичном координационом окружењу. Дисиликатне групе Si_2O_7 повезују четири EuO_6 октаедра и једну EuO_6 тригоналну призму. Између њих се налазе три различито координисана катјона калијума. Силикати који садрже веће јоне ретких земаља имају структуру која садржи катјон ретке земље у два различита координациона окружења: у благо искривљеном октаедру и у идеалном тригонално-призматичном окружењу.

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