

GEOCHEMISTRY

# Crystal Structure of Hyperzirconium Sulfate Analogue of Eudialyte

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Eudialyte is the common mineral of alkaline apgaitic massifs. In some cases, it occurs as a major mineral and important carrier of some rare elements. The crystal chemical formula of typical eudialyte contains six Ca (as a six-membered octahedral ring) and three Zr atoms p.f.u. However, eudialytes formed in specific conditions have different stoichiometry. For example, hypercalcium analogues of eudialyte (feklichchevite [1], golyshevite [2], mogovidite [3]) were found in pegmatites related to alkaline ultrabasic rocks. These minerals typically contain 8–10 Ca atoms p.f.u., with excess Ca incorporated in one or two extraframework sites. By contrast, eudialyte analogues (oneillite [4], raslakite [5], and low-symmetry analogues [6–8]) containing only ~3 Ca atoms p.f.u. were formed under Ca deficiency in some apgaitic pegmatites. In this case, Ca, Mn, Fe, and Na can be ordered in an octahedral ring with the symmetry degradation from  $R3m$  to  $R3$ .

The mineral studied in this work is also depleted in Ca. However, unlike oneillite and raslakite, the studied mineral is also enriched in Zr content ( $\text{Ca}/\text{Zr} \sim 0.6$ ). Such a ratio was found in the sample reported in [7]. In addition, the mineral also contains a significant amount of sulfur.

This eudialyte-group mineral was found in ultraagpaitic pegmatite on Mt. Alluaiv (Lovozero Massif, Kola Peninsula). Its empirical formula ( $Z = 3$ ) based on microprobe analysis and the consideration of grain heterogeneity is as follows:  $\text{Na}_{14-17} \text{K}_{0.2-0.4} \text{Sr}_{0-0.6} \text{REE}_{0.2-0.6} \text{Ca}_{2.6-2.9} \text{Zr}_{3.9-4.8} \text{Fe}_{0.4-1.3} \text{Mn}_{1.5-1.8} \text{Ti}_{0.2-0.4} \text{Nb}_{0-0.6} \text{Si}_{25} \text{S}_{0.3-0.5} \text{Cl}_{0.2-0.5} \text{F}_{0.4} (\text{O}, \text{OH})_x \cdot n\text{H}_2\text{O}$ , where  $x \sim 75$  and  $n \sim 1$ .

Characteristics of the crystal and experimental data are given in Table 1.

According to the accepted nomenclature, eudialyte-group minerals can be described by the general formula:

$[A(1)A(2)A(3)A(4)A(5)A(6)A(7)]_3A(8)[M(1.1)M(1.2)]_3[M(2.1)M(2.2)M(2.3)]_{3-6}[M(3)M(4)]Z_3[\text{Si}_{24}\text{O}_{72}]\text{OH}_{2-6}\text{X}_{2-4}$ , where  $A = \text{Na}, \text{Ca}, \text{K}, \text{Sr}, \text{REE}, \text{Ba}, \text{Mn}, \text{H}_3\text{O}$ ;  $M(1.1), M(1.2) = \text{Ca}, \text{Mn}, \text{REE}, \text{Na}, \text{Sr}, \text{Fe}$ ;  $M(2.1) = \text{Fe}, \text{Na}, \text{Zr}, \text{Ta}$ ;  $M(2.2)$  and  $M(2.3) = \text{Fe}, \text{Mn}, \text{Zr}, \text{Ti}, \text{Na}, \text{K}, \text{Ba}, \text{H}_3\text{O}$ ;  $M(3)$  and  $M(4) = \text{Si}, \text{Nb}, \text{Ti}, \text{W}, \text{Na}$ ;  $Z = \text{Zr}, \text{Ti}$ ;  $X = \text{H}_2\text{O}, \text{Cl}, \text{F}, \text{OH}, \text{CO}_3, \text{SO}_4, \text{AlO}_4$ . It should be noted that not all of the sites are completely occupied. In particular, only the first four  $A$  sites are occupied completely by Na and substituting elements, while  $A(5)$ – $A(8)$  can be

Table 1. Data on crystal and experimental conditions

Unit cell parameters, Å	$a = 14.192(5)$ $c = 30.20(1)$
Unit cell volume, Å <sup>3</sup>	$V = 5267.75$
Space group	$R3$
Radiation; $\lambda$ , Å	$\text{MoK}\alpha$ ; 0.71073
Density calc., g/cm <sup>3</sup>	2.88
Crystal size, mm	$0.2 \times 0.15 \times 0.10$
Diffractometer	ENRAF–NONIUS
$h, k, l$ limits	$-19 < h < 17$ $0 < k < 19$ $0 < l < 42$
$\sin\theta/\lambda$	$< 0.702$
Total number of reflections	$4100 F > 5\sigma(F)$
Number of independent reflections	$2455 F > 5\sigma(F)$
$R$ -factor of averaging of the equivalent reflections	0.039
$R$ -factor of anisotropic refinement	0.049
Refinement program	AREN [9]
Program for absorption correction	DIFABS[10]

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**Table 2.** Coordinates and equivalent parameters of thermal displacements of the framework atoms

Atom	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	<i>B</i> <sub>equiv</sub> , Å <sup>2</sup>
Zr	0.3335(1)	0.1662(1)	0.1666(1)	1.66(3)
Si(1)	0.9915(1)	0.6040(2)	0.0961(1)	1.1(1)
Si(1)'	0.9927(1)	0.3901(2)	0.0970(1)	1.1(1)
Si(2)	0.1394(2)	0.0665(2)	0.0812(1)	1.5(1)
Si(3)	0.0561(2)	0.3257(2)	0.2362(1)	1.2(1)
Si(3)'	0.2710(2)	0.3249(2)	0.2371(1)	1.2(1)
Si(4)	0.2081(2)	0.4156(2)	0.0761(1)	1.3(1)
Si(5)	0.5254(2)	0.2593(2)	0.2522(1)	1.6(1)
Si(6)	0.4578(2)	0.5410(2)	0.2581(1)	1.5(1)
O(1)	0.1796(7)	0.3595(6)	0.0291(2)	2.3(4)
O(2)	0.1789(6)	0.3555(6)	0.2205(2)	2.0(4)
O(3)	0.6222(7)	0.5895(6)	0.0450(2)	2.4(4)
O(3)'	0.6323(6)	0.0367(6)	0.0463(2)	1.9(4)
O(4)	0.2577(6)	0.0260(6)	0.2064(3)	2.3(4)
O(4)'	0.2620(8)	0.2314(6)	0.2072(3)	2.8(4)
O(5)	0.4743(6)	0.2319(8)	0.2043(2)	2.6(4)
O(6)	0.2262(6)	0.1046(9)	0.0416(3)	2.9(4)
O(7)	0.1942(8)	0.0904(9)	0.1294(3)	3.5(4)
O(8)	0.1038(5)	0.3858(6)	0.1069(2)	1.8(4)
O(8)'	0.2771(6)	0.3788(7)	0.1080(2)	1.9(4)
O(9)	0.4446(8)	0.213(1)	0.2920(3)	3.6(4)
O(10)	0.6035(6)	0.2097(6)	0.2561(3)	2.4(4)
O(11)	0.0597(6)	0.1225(5)	0.0780(3)	2.3(4)
O(12)	0.0458(7)	0.4323(6)	0.2278(3)	2.4(4)
O(12)'	0.3908(6)	0.4313(7)	0.2265(2)	2.4(4)
O(13)	0.0264(6)	0.5119(6)	0.3046(2)	2.0(4)
O(14)	0.4086(7)	0.3022(7)	0.1280(2)	2.5(4)
O(14)'	0.4121(6)	0.1057(7)	0.1287(2)	2.2(4)
O(15)	0.0400(7)	0.2964(6)	0.2890(2)	2.2(4)
O(15)'	0.2626(6)	0.2940(6)	0.2880(2)	1.8(4)
O(16)	0.0215(6)	0.5109(5)	0.1122(2)	1.8(4)
O(17)	0.2713(7)	0.5428(7)	0.0739(5)	4.5(4)
O(18)	0.391(1)	0.606(1)	0.2614(5)	4.8(2)

either partially randomly occupied or left completely vacant.

The structural closeness to raslakite [5] made it possible to use its structure framework for refining the structure of the studied mineral. Other sites were found from the electron density difference map. The coordinates of atoms and characteristics of coordination polyhedra for the studied mineral are listed in Tables 2–4.

The structure of the mineral is generally similar to those of other eudialyte-group minerals. It includes three- and nine-membered rings of Si–O tetrahedra and six-membered rings of (Ca,O) octahedra combined into

a heterogeneous framework by discrete (Zr,O) octahedra. The framework cavities contain cations of alkali, alkali earth, and transition metals, as well as large additional anions.

The main compositional and structural features of the mineral are reflected in its crystallochemical formula ( $Z = 3$ ), which is well consistent with the empirical one  $(\text{Na}_{13.66}\text{K}_{0.22}\text{Sr}_{0.48}\text{REE}_{0.16})[(\text{Ca}_{2.85}\text{REE}_{0.15})(\text{Mn}_{1.8}\text{Fe}_{1.2})]\text{Zr}_3[\text{Si}_{24}\text{O}_{72}][(\text{Zr}_{0.81}\text{Zr}_{0.66}^{\text{IV}})\text{Na}_{0.96}^{\text{VI}}][\text{Si}_{0.5}\text{Nb}_{0.3}\text{Si}_{0.2}][\text{S}_{0.4}(\text{Ti}_{0.3}\text{Si}_{0.3})\text{O}_{1.0}(\text{O}, \text{OH})_{2.02}(\text{OH}, \text{H}_2\text{O})_{1.7}\text{Cl}_{0.5}\text{F}_{0.4}]$ , where brackets designate the key sites of the structure, while Roman numerals denote the coordination numbers of cations. The idealized formula can be presented as follows ( $Z = 3$ ):  $\text{Na}_{15}[\text{Ca}_3(\text{Mn}, \text{Fe})_3]\text{Zr}_3[\text{Zr}, \text{Na}]_3[\text{Si}, \text{Nb}]_1[\text{S}, \text{Ti}, \text{Si}]_1[\text{Si}_3\text{O}_9]_2[\text{Si}_9\text{O}_{27}]_2(\text{O}, \text{OH})_5(\text{Cl}, \text{F}, \text{H}_2\text{O})_1$ .

It was found that the six-membered ring contains two independent and contrasting (in volume) octahedra. One of them is dominated by Ca, while the other is dominated by Mn. The average  $M(1,2)\text{--O}$  and  $M(1,1)\text{--O}$  values are 2.35 and 2.29 Å, respectively.

The tetrahedral and octahedral  $M$  sites are localized near the center of both nine-membered rings of Si–O tetrahedra.

The  $M(4)$  site is split into three subsites, which are statistically occupied by Nb and Si. The  $M(4a)$  subsite makes up Nb octahedra with a Nb–O distance of 1.82–1.87 Å. The triangular oxygen base of this octahedron is shared by two oppositely oriented Si tetrahedra, with the average Si–O distance of 1.60 and 1.63 Å, respectively. The  $M(4a)$  and  $M(4b)$  subsites are spaced at 0.51 Å, while Si sites in  $M(4b)$  and  $M(4c)$  are spaced at 0.95 Å.

The  $M(3)$  site is split into two subsites, which are spaced at 1.3 Å from each other and statistically occupied by Ti, Si, and S, respectively. The  $M(3a)$  subsite is equally shared by Ti and Si, while the  $M(3b)$  site is occupied by S. The sites surrounding the mixed  $M(3a)$  subsite are arranged into an oxygen triangle of framework Si tetrahedra, with three additional O (19) around axis 3 and an (O,OH)(2) group on the axis, so that Ti occurs in the center of an octahedron with a Ti–O distance of 1.67–1.77 Å, while Si is in tetrahedral coordination with an average Si–O distance of 1.63. The third polyhedron  $M(3b)$ —the sulfur tetrahedron with an average S–O distance of 1.52 Å—is located inside the cavity between rings. The apical vertex of sulfur tetrahedra is supposed to be occupied by 0.4 F atoms, which is equal to the amount of sulfur. In [11], the sulfur tetrahedron (0.2 atoms) was found among X anions in equal proportions with Cl and H<sub>2</sub>O. The allocation of SO<sub>4</sub> groups to the center of a nine-membered ring required additional checkup of the local balance of oxygen atoms shared between Si and S tetrahedra. The bond strength on a Si(6)–O(18)–S bridging atom is 2.3, which slightly exceeds the value of 10% accepted as the permissible deviation from 2. However, this deviation can be considered justified because the local balance is

**Table 3.** Coordinates and equivalent parameters ( $B_{\text{equiv}}$ ) of thermal displacements of intraframework atoms and the multiplicity ( $Q$ ) and occupancy ( $q$ ) of sites

Atom	$x/a$	$y/b$	$z/c$	$B_{\text{equiv}}, \text{\AA}^2$	$Q$	$q$
$M(1.1)$	-0.0002(1)	0.2652(1)	0.0002(1)	1.50(2)	9	1
$M(1.2)$	0.2593(1)	0.2592(1)	0.0002(1)	1.49(2)	9	1
$A(1a)$	0.1085(5)	0.2185(5)	0.1537(1)	2.78(9)	9	0.72(1)
$A(1b)$	0.082(1)	0.172(2)	0.1687(6)	4.8(3)	9	0.28(1)
$A(2a)$	0.5527(4)	0.4488(4)	0.1813(2)	2.5(1)	9	0.79(1)
$A(2b)$	0.589(2)	0.428(2)	0.169(1)	9.6(3)	9	0.23(2)
$A(3a)$	0.1958(5)	0.0987(4)	0.2887(2)	3.23(7)	9	0.53(1)
$A(3b)$	0.234(1)	0.118(1)	0.2794(3)	3.2(2)	9	0.47(1)
$A(4a)$	0.4538(7)	0.2267(6)	0.0469(2)	2.1(1)	9	0.54(1)
$A(4b)$	0.441(1)	0.220(1)	0.0617(8)	3.9(3)	9	0.30(1)
$A(4c)$	0.4796(5)	0.2420(5)	0.0392(3)	4.4(2)	9	0.16(1)
$A(5a)$	0.185(1)	0.580(2)	0.1508(7)	6.6(7)	9	0.42(3)
$A(5b)$	0.246(2)	0.522(1)	0.1823(7)	6.4(5)	9	0.42(3)
$A(6)$	0.113(1)	0.227(1)	0.3376(6)	4.4(1)	9	0.32(1)
$M(3a)$	0.3333	0.6666	0.2892(3)	2.8(1)	3	0.60(1)
$M(3b)$	0.3333	0.6666	0.2460(6)	3.9(5)	3	0.40(2)
$M(4a)$	0.3333	0.6666	0.0403(1)	1.90(6)	3	0.30(1)
$M(4b)$	0.3333	0.6666	0.0572(5)	2.2(3)	3	0.20(1)
$M(4c)$	0.3333	0.6666	0.0887(2)	1.6(9)	3	0.50(1)
$M(2.1)$	0.4955(5)	0.5035(5)	0.0027(2)	2.02(4)	9	0.27(1)
$M(2.2)$	0.5208(3)	0.4787(3)	-0.0032(1)	2.14(4)	9	0.22(1)
(O, OH)(1)	0.3333	0.6666	0.003(6)	3.2(9)	3	0.32(4)
(O, OH)(2)	0.6666	0.3333	0.006(2)	4.1(6)	3	0.30(6)
(O, OH)(3)	0.3333	0.6666	0.147(1)	2.6(1)	3	0.50(1)
(O, OH)(4)	0.393(2)	0.599(2)	0.006(1)	3.3(5)	9	0.30(1)
F	0.3333	0.6666	0.195(2)	2.8(8)	3	0.40(1)
Cl	0	0	0.2899(6)	5.0(4)	3	0.50(3)
O(19)	0.612(3)	0.384(3)	-0.004(1)	5.6(4)	9	0.30(1)
(OH, H <sub>2</sub> O)(1)	0.6666	0.3330	0.066(7)	4.2(5)*	3	0.30(6)
(OH, H <sub>2</sub> O)(2)	0.6666	0.3333	0.096(2)	4.7(7)	3	0.35(4)
(OH, H <sub>2</sub> O)(3)	0	0	0.207(2)	8.9(7)	3	0.39(5)
(OH, H <sub>2</sub> O)(4)	0	0	0.229(1)	3.1(9)	3	0.50(8)
(OH, H <sub>2</sub> O)(5)	0	0	0.185(5)	5(2)*	3	0.16(4)

Notes: \* Isotropic thermal parameters.

regulated by the Si(6)–O(18) bond length. However, the bond in this structure is shortened due to the forbidden splitting of O(18) site with thermal parameter  $4.8 \text{ \AA}^2$  (Table 2) at statistical occupancy of related S, Si, and Ti sites.

Zr is mainly allocated in the regular octahedra with  $Zr-O = 2.042-2.096 \text{ \AA}$ . The Zr atoms in excess of 1.47 occupy  $M(2)$  sites. The attempts to allocate Zr in axial  $M(3)$  and  $M(4)$  polyhedra failed because the thermal parameter in the  $M(3)$  site seemed to be very high,

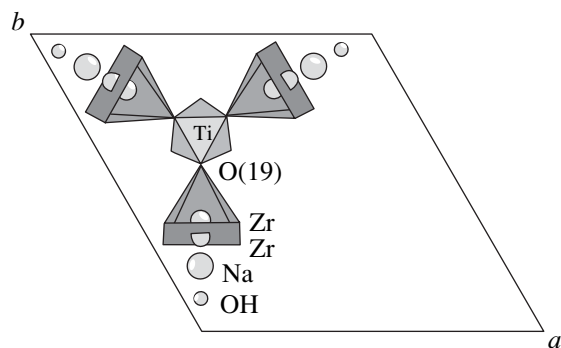
while both octahedra appeared to be too small for Zr: the Ti–O and Nb–O distances are  $1.67-1.77$  and  $1.82-1.87 \text{ \AA}$ , respectively. However, the “square” center site was approximated by Fe atoms and the thermal parameter took a negative value, requiring the presence of a heavier atom. An additional peak obtained by an electron density difference map at a distance of  $0.64 \text{ \AA}$  from the square center also required approximation by a cation heavier than Fe. The refinement of the thermal parameter for both sites with Zr atoms decreased the  $R$  factor and

**Table 4.** Characteristics of coordination polyhedra

Site	Composition (Z = 3)	CN	Cation-anion distance, Å		
			min	max	av
Zr	3Zr	6	2.042(9)	2.096(8)	2.066
M(1.1)	1.8Mn + 1.2Fe	6	2.211(5)	2.38(1)	2.29
M(1.2)	2.85Ca + 0.15Ce	6	2.297(6)	2.38(1)	2.35
A(1a)	1.94Na + 0.22K	7	2.56(1)	2.79(1)	2.66
A(1b)	0.84Na	6	2.37(2)	2.81(2)	2.60
A(2a)	2.37Na	8	2.54(1)	2.79(1)	2.62
A(2b)	0.63Na	6	2.33(3)	2.82(4)	2.57
A(3a)	1.43Na + 0.16Ce	5	2.43(1)	2.98(1)	2.69
A(3b)	1.41Na	6	2.34(1)	2.66(1)	2.55
A(4a)	1.62Na	6	2.42(1)	2.89(1)	2.67
A(4b)	0.9Na	6	2.44(2)	2.89(2)	2.58
A(4c)	0.48Sr	6	2.50(1)	3.17(1)	2.84
A(5a)	1.26Na	6	2.32(2)	3.10(3)	2.82
A(5b)	1.26Na	5	2.36(2)	3.01(2)	2.79
A(6)	0.96Na	6	2.28(2)	3.14(2)	2.64
M(2.1)	0.81Zr	4	2.03(1)	2.22(1)	2.13
M(2.2)	0.66Zr	5	2.099(8)	2.302(5)	2.217
M(3a)	0.3Ti + 0.3Si	6	1.67(1)	1.77(2)	1.72
		4	1.52(4)	1.67(1)	1.63
M(3b)	0.4S	4	1.52(3)	1.53(3)	1.52
M(4a)	0.3Nb	6	1.82(1)	1.87(3)	1.85
M(4b)	0.2Si	4	1.60(1)	1.60(1)	1.60
M(4c)	0.5Si	4	1.59(3)	1.76(1)	1.63

Note: Si-tetrahedra of the framework are omitted. Randomly occupied anion sites in A polyhedra, except A(6), were not included.

yielded normal values of thermal and other characteristics. Thus, the additional Zr atoms are located in two subsites with total occupancy of 1.47 atoms p.f.u.: in the M(2,1) "square" with distances of 2.03–2.22 Å (aver-



Fragment of the structure of a new eudialyte analogue in the (001) projection as a cluster of three Zr-pentahedra and a central Ti octahedron.

**Table 5.** Geometric parameters of "squares" for various M(2) cations

Atom	Distance M(2)–O, Å	Distance O–O, Å	Reference
Fe	2.03(1)–2.06(1)	2.95, 2.98, 2.80 × 2	[14]
Zr	2.03(1)–2.22(1)	2.83, 2.92, 3.11, 3.17	This work
Zr	2.04(1)–2.12(1)	2.78, 2.81, 3.03, 3.08	[5]
Ta	2.04(1)–2.12(1)	2.95, 3.11, 2.85 × 2	[15]
Na	2.29(1)–2.29(1)	3.28, 3.28, 3.13 × 2	[16]

age 2.13 Å) and in the M(2, 2) pentahedron with distances of 2.10–2.30 Å (average 2.22 Å). As a result, Zr in the given fragment prevails over A(6), which is allocated in the M(2.3) subsites occupied by 0.96 Na atoms on the opposite side of the square at a distance of 1.21 Å. The total occupancy of cations (Zr and Na) in the given fragment is less than three, because the (O,OH)(4) group from sites surrounding Nb is closely spaced (1.2 Å) from Na site A(6) and does not participate in its polyhedron (figure).

The Zr pentahedra reported in [6, 7] are discrete and they have no contact with axial octahedra. In the present structure, the Zr pentahedra are bound through O(19) with the Ti octahedron on the axis 3, and they make up a cluster (figure). Such a cluster was previously found in a hyperzirconium analogue of eudialyte [12]. However, excess atoms of Zr (1.2) in this mineral were allocated in an octahedron together with a minor Fe, and its most part (1 atom) was localized together with Ca in the octahedron of a six-membered ring.

Previously, we found tetracoordinated Zr in raslakite [5]. As in the present mineral, all Fe (2.2 atoms) was released from its typical site into the Ca octahedron, where it substitutes Ca together with Mn and Ce.

Recent X-ray analysis has shown that the square position in eudialytes can be occupied by not only Fe, but also Nb, Ta, and Zr. The square size depends only on cation type. Table 5 shows the geometric parameters of squares for various M(2) elements.

Thus, the new eudialyte-group mineral is closest to raslakite. The mineral is characterized by a predominance of Zr in M(2) sites and elevated content of sulfate S detected for the first time in the M(3) axial site (instead of anion X group), where S dominates over Ti and Si.

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