Crystal structure of tetraaquatrissuccinatodierbium hexahydrate, $\text{Er}_2(\text{H}_2\text{O})_4(\text{C}_4\text{H}_4\text{O}_4)_3 \cdot 6\text{H}_2\text{O}$

J. Sun, Y.-Q. Zheng* and J.-L. Lin

Ningbo University, Institute for Solid State Chemistry, Municipal Key Laboratory of Inorganic Materials Chemistry, Ningbo, Zhejiang, 315211 P. R. China

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Abstract

$\text{C}_{12}\text{H}_{32}\text{Er}_2\text{O}_{22}$, triclinic, $P\bar{1}$ (no. 2), $a = 6.517(1)$ Å, $b = 9.774(1)$ Å, $c = 10.466(1)$ Å, $\alpha = 80.19(1)^\circ$, $\beta = 89.84(1)^\circ$, $\gamma = 71.37(1)^\circ$, $V = 621.5$ Å³, $Z = 1$, $R_{\text{gt}}(F) = 0.031$, $wR_{\text{ref}}(F^2) = 0.080$, $T = 293$ K.

Source of material

A 23 ml Teflon-lined stainless-steel vessel charged with a mixture of 0.10 g (0.26 mmol) Er_2O_3 and 0.19 g (1.57 mmol) succinic acid in 15 ml H_2O was heated at 160 °C for 10 days. After the vessel was cooled to room temperature, a small amount of reddish crystals was separated by filtration.

Discussion

The title compound consists of hydrate water molecules and 2D layers formulated as $[\text{Er}_2(\text{H}_2\text{O})_4(\text{C}_4\text{H}_4\text{O}_4)_3]$. This is in contrast to the recently reported lutetium(III) succinate hydrate, in which the eight-coordinated Lu atoms are bridged by *anti*- and *gauche* bis-bidentate succinate anions into 3D framework with water molecules in cavities [1]. The Er atoms are nine-fold coordinated by seven oxygen atoms of four succinate anions and two oxygen atoms of different aqua ligands to form $[\text{ErO}_9]$ monocapped square antiprisms with $d(\text{Er}—\text{O}) = 2.302$ Å – 2.586 Å. Two adjacent $[\text{ErO}_9]$ polyhedra are edge-shared to generate a $[\text{Er}_2\text{O}_{16}]$ bi-polyhedron centered at the crystallographic $1h$ site. The Er...Er separation is of 4.105 Å within the bi-polyhedron. Two crystallographically distinct succinate anions have the expected bonding values [1–5]. The *gauche* one, with a torsion angle of -62.2° , functions as a penta-dentate ligand bischelating two Er atoms

with one chelating oxygen atom bonding to the third Er atom. The *anti* conformational one with the coplanar non-hydrogen atoms bischelates two Er atoms. Along the $[100]$, the bi-polyhedra are bridged by the twisted succinato groups into ribbon-like chains, which are further interconnected by the bischelating succinato groups to form open layers parallel to (010) . The water molecules are located between the resulting layers. In the structure extensive hydrogen bonds exist: (i) between aqua ligand and lattice H_2O molecule with $d(\text{O} \cdots \text{O}) = 2.682$ Å, 2.739 Å, (ii) between lattice H_2O molecules themselves with $d(\text{O} \cdots \text{O}) = 2.735$ Å – 2.928 Å and (iii) between lattice H_2O molecule and carboxylate oxygen atom with $d(\text{O} \cdots \text{O}) = 2.747$ Å – 3.071 Å, and (iv) intralayer hydrogen bonds between aqua ligand and carboxylate oxygen atom with $d(\text{O} \cdots \text{O}) = 2.664$ Å, 2.798 Å.

Table 1. Data collection and handling.

Crystal:	pale red block, size $0.044 \times 0.089 \times 0.267$ mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	68.01 cm^{-1}
Diffractometer, scan mode:	Bruker P4, $\theta/2\theta$
$2\theta_{\text{max}}$:	55°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	3600, 2848
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2630
$N(\text{param})_{\text{refined}}$:	165
Programs:	SHELXS-97 [6], SHELXL-97 [7]

* Correspondence author (e-mail: zhengcm@nbu.edu.cn)

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U _{iso}
H(2A)	2i	0.8161	0.7037	0.6493	0.061(9)
H(2B)	2i	0.6143	0.8455	0.6439	0.061
H(3A)	2i	0.7331	0.9560	0.4533	0.061
H(3B)	2i	0.9062	0.9165	0.5688	0.061
H(6A)	2i	0.1437	0.3592	0.0512	0.061
H(6B)	2i	0.2049	0.4722	−0.0535	0.061
H(7A)	2i	0.3962	0.7602	0.0702	0.050
H(7B)	2i	0.4917	0.8189	0.1244	0.050

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(8A)	2i	0.8408	0.5430	0.2696	0.050
H(8B)	2i	0.8331	0.4575	0.3659	0.050
H(9A)	2i	0.1835	0.8202	−0.1243	0.050
H(9B)	2i	0.3788	0.7305	−0.1460	0.050
H(10A)	2i	0.6927	1.0009	0.1254	0.050
H(10B)	2i	0.8510	0.8618	0.1411	0.050
H(11A)	2i	1.1634	1.0254	0.2080	0.050
H(11B)	2i	1.1672	1.0985	0.0759	0.050

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Er	2i	0.37408(3)	0.62386(2)	0.32213(2)	0.0268(1)	0.0311(1)	0.0245(1)	−0.01220(9)	0.00140(7)	−0.00551(8)
O(1)	2i	0.5466(6)	0.6072(4)	0.5489(3)	0.039(2)	0.034(2)	0.039(2)	−0.017(1)	0.009(1)	−0.010(1)
O(2)	2i	0.5231(7)	0.7919(4)	0.3949(4)	0.059(2)	0.041(2)	0.038(2)	−0.024(2)	−0.013(2)	−0.002(2)
C(1)	2i	0.5886(7)	0.7251(5)	0.5066(5)	0.024(2)	0.029(2)	0.036(2)	−0.010(2)	0.004(2)	−0.010(2)
C(2)	2i	0.7138(8)	0.7847(6)	0.5912(4)	0.034(2)	0.041(2)	0.029(2)	−0.019(2)	0.007(2)	−0.012(2)
C(3)	2i	0.8364(8)	0.8751(6)	0.5109(5)	0.035(2)	0.035(2)	0.046(3)	−0.019(2)	0.013(2)	−0.018(2)
C(4)	2i	1.0055(7)	0.7890(5)	0.4309(4)	0.026(2)	0.039(2)	0.030(2)	−0.015(2)	0.002(2)	−0.013(2)
O(3)	2i	1.0901(6)	0.6511(4)	0.4674(3)	0.034(2)	0.036(2)	0.035(2)	−0.006(1)	0.007(1)	−0.001(1)
O(4)	2i	1.0668(7)	0.8510(4)	0.3314(4)	0.046(2)	0.037(2)	0.041(2)	−0.018(2)	0.014(2)	−0.007(2)
O(5)	2i	0.0743(6)	0.6147(5)	0.1869(4)	0.029(2)	0.054(2)	0.038(2)	−0.016(2)	0.002(1)	−0.022(2)
O(6)	2i	0.4005(6)	0.4768(4)	0.1583(4)	0.030(2)	0.050(2)	0.041(2)	−0.011(2)	−0.001(1)	−0.019(2)
C(5)	2i	0.2007(8)	0.5223(5)	0.1272(5)	0.035(2)	0.036(2)	0.029(2)	−0.017(2)	−0.000(2)	−0.009(2)
C(6)	2i	0.1193(8)	0.4630(7)	0.0214(5)	0.037(3)	0.052(3)	0.040(3)	−0.013(2)	−0.003(2)	−0.022(2)
O(7)	2i	0.4173(6)	0.7778(4)	0.1391(3)	0.044(2)	0.047(2)	0.030(2)	−0.022(2)	0.003(1)	−0.003(1)
O(8)	2i	0.7477(6)	0.5172(4)	0.3043(3)	0.029(2)	0.050(2)	0.032(2)	−0.014(2)	0.003(1)	0.000(1)
O(9)	2i	0.3221(8)	0.7857(5)	−0.1118(4)	0.058(3)	0.057(3)	0.049(2)	−0.011(2)	0.002(2)	−0.021(2)
O(10)	2i	0.7257(7)	0.9147(5)	0.1083(5)	0.048(2)	0.052(2)	0.059(3)	−0.022(2)	−0.008(2)	0.006(2)
O(11)	2i	1.1191(9)	1.1060(5)	0.1528(5)	0.070(3)	0.046(2)	0.062(3)	−0.014(2)	0.003(2)	−0.006(2)

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