Crystal structure of dieuropium trisulfate octahydrate, $\text{Eu}_2(\text{SO}_4)_3 \cdot 8\text{H}_2\text{O}$

D.-Y. Wei and Y.-Q. Zheng*

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

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Abstract

$\text{Eu}_2\text{H}_{16}\text{O}_{20}\text{S}_3$, monoclinic, $C12/c1$ (No. 15), $a = 13.555(2)$ Å, $b = 6.757(1)$ Å, $c = 18.317(2)$ Å, $\beta = 102.27(1)^\circ$, $V = 1639.4$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.042$, $wR_{\text{ref}}(F^2) = 0.105$, $T = 293$ K.

Source of material

0.10 g Eu_2O_3 was wetted with doubly distilled water and then a small amount of concentrated H_2SO_4 was dropwise added until the solid was completely dissolved. The resulting solution was subsequently heated nearly to dryness. After the obtained crystalline substance was dissolved in 20 ml $\text{CH}_3\text{OH}/\text{H}_2\text{O}$ (1:1 v/v), 0.09 g phenanthroline was added with stirring. The mixture was further stirred for one hour and then allowed to stand at room temperature. Nearly colorless plate-like crystals were grown by slow evaporation at room temperature for four weeks.

Discussion

The title compound crystallizes isostructurally with $\text{Pr}_2(\text{SO}_4)_3 \cdot 8\text{H}_2\text{O}$ [1] and the Eu atoms are each located in an antiprismatic coordination geometry of four aqua ligands, one $\mu_2\text{-SO}_4^{2-}$ and three $\mu_3\text{-SO}_4^{2-}$ anions with $d(\text{Eu}-\text{O}) = 2.337$ Å – 2.511 Å. Actually, the water O atoms and the sulfato O atoms around the central Eu atom, respectively, occupy the corners of two interpenetrating tetrahedra. The $\mu_2\text{-SO}_4^{2-}$ anions are on the crystallographic C_2 symmetry with the mean $d(\text{S1}-\text{O}) = 1.467(5)$ Å and $\angle\text{O}-\text{S1}-\text{O} = 108.8^\circ - 110.6^\circ$, whereas the $\mu_3\text{-SO}_4^{2-}$ group with $d(\text{S2}-\text{O}) = 1.455$ Å – 1.492 Å and $\angle\text{O}-\text{S2}-\text{O} = 107.2^\circ - 111.8^\circ$ exhibits significant deviation from the ideal T_d symmetry. Along [010] direction, the Eu atoms are bridged by $\mu_3\text{-SO}_4^{2-}$ groups into ribbon-like chains which are further bridged by $\mu_2\text{-SO}_4^{2-}$ groups along the [101] direction to form two-dimensional $[\text{Eu}(\text{H}_2\text{O})_4(\mu_2\text{-SO}_4\text{-O}, \text{O}')_{1/2}(\mu_3\text{-SO}_4\text{-O}, \text{O}', \text{O}'')_{3/3}]$ layers parallel to (101) with 8- and 16-membered rings. The water molecules donate hydrogen atoms to sulfato O atoms and water O atoms to form intra- and interlayer hydrogen bonds with $d(\text{O}\cdots\text{O}) = 2.721$ Å – 3.024 Å and $\angle\text{O}-\text{H}\cdots\text{O} = 149^\circ - 173^\circ$. The 2D layers are held together by the interlayer hydrogen bonds.

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Table 1. Data collection and handling.

Crystal:	colorless, plate-like, size $0.067 \times 0.178 \times 0.556$ mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	80.66 cm ⁻¹
Diffractometer, scan mode:	Bruker P4, $\theta/2\theta$
$2\theta_{\text{max}}$:	55°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	2481, 1881
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1754
$N(\text{param})_{\text{refined}}$:	115
Program:	SHELXL-97 [2]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	8f	-0.0513	-0.1190	0.3507	0.05
H(1B)	8f	0.0058	-0.2903	0.3448	0.05
H(2A)	8f	0.3236	-0.0285	0.3029	0.05
H(2B)	8f	0.2339	-0.0150	0.2449	0.05
H(3A)	8f	0.3798	0.0919	0.4835	0.05
H(3B)	8f	0.3556	-0.1330	0.4795	0.05
H(4A)	8f	0.0163	0.3306	0.4099	0.05
H(4B)	8f	-0.0078	0.1826	0.4485	0.05

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

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
Eu	8f	0.16721(2)	0.02205(4)	0.39204(1)	0.0216(2)	0.0185(2)	0.0214(2)	0.00120(7)	0.0043(1)	0.00059(7)
O(1)	8f	0.0142(3)	−0.1665(6)	0.3592(3)	0.027(2)	0.025(2)	0.066(3)	0.000(2)	0.001(2)	−0.007(2)
O(2)	8f	0.2577(4)	−0.0105(7)	0.2960(2)	0.031(2)	0.064(3)	0.022(2)	0.016(2)	0.010(2)	0.007(2)
O(3)	8f	0.3449(3)	−0.0154(5)	0.4565(3)	0.026(2)	0.026(2)	0.029(2)	0.003(1)	0.003(2)	−0.002(1)
O(4)	8f	0.0396(2)	0.2345(6)	0.4363(2)	0.025(2)	0.029(2)	0.030(2)	0.003(1)	0.006(1)	0.004(1)
S(1)	4e	0	0.3234(3)	1/4	0.0215(7)	0.0217(8)	0.0218(7)	0	0.0028(5)	0
O(5)	8f	0.0843(3)	0.1991(7)	0.2868(2)	0.035(2)	0.039(2)	0.028(2)	0.013(2)	0.007(1)	0.004(2)
O(6)	8f	−0.0334(3)	0.4474(6)	0.3062(2)	0.031(2)	0.025(2)	0.032(2)	−0.000(2)	0.007(2)	−0.008(2)
S(2)	8f	0.21717(9)	−0.0287(2)	0.58897(7)	0.0219(6)	0.0184(6)	0.0217(6)	0.0004(4)	0.0047(5)	−0.0004(4)
O(7)	8f	0.1466(3)	−0.0777(6)	0.5170(2)	0.029(2)	0.027(2)	0.026(2)	0.000(2)	0.003(1)	−0.001(2)
O(8)	8f	0.2587(3)	0.1670(6)	0.5804(2)	0.037(2)	0.023(2)	0.043(2)	−0.004(2)	0.008(2)	0.003(2)
O(9)	8f	0.2994(3)	−0.1754(6)	0.6024(2)	0.025(2)	0.023(2)	0.033(2)	0.003(2)	0.005(1)	−0.001(1)
O(10)	8f	0.1621(3)	−0.0330(7)	0.6491(2)	0.028(2)	0.043(2)	0.022(2)	0.003(2)	0.010(2)	0.002(1)

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