inorganic papers

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(S–O) = 0.002 Å
R factor = 0.019
wR factor = 0.054
Data-to-parameter ratio = 16.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Dieuropium(III) trisulfate octahydrate: a redetermination at 120 K

The title compound, $Eu_2(SO_4)_3 \cdot 8H_2O$, crystallizes in space group $C2/c$, with one of the anions lying on a twofold rotation axis and the other in a general position, and is best formulated as $[Eu(H_2O)_{4/1}(SO_4)_{3/3}(SO_4)_{1/2}]_2$, where one of the anions lies across a twofold axis. The coordination environment of Eu^{III} consists of four water molecules and four sulfate ions. All the water molecules and sulfate ions are involved in hydrogen-bonding interactions. The structure is similar to that previously determined at 293 K [Wei & Zheng (2003). *Z. Kristallogr. New Cryst. Struct.* **218**, 277–278], but the cell parameters and the interatomic distances are more precise in the present determination.

Comment

Hydrated lanthanide(III) sulfates can adopt a number of different compositions, namely $M_2(SO_4)_3 \cdot 9H_2O$, $M_2(SO_4)_3 \cdot 8H_2O$, $M_2(SO_4)_3 \cdot 5H_2O$ and $M_2(SO_4)_3 \cdot 4H_2O$, and the octahydrated sulfates of lanthanides Ln^{III} exist as coordination polymers in which sulfate ions act as bridging bidentate and tridentate ligands; the presence of four coordinated water molecules leads to a coordination number of eight for the lanthanide ion (Wickleder, 2002). The unit-cell dimensions for hydrated europium(III) sulfate were reported many years ago (Geller, 1957), and the crystal structure, in space group $C2/c$, has recently been reported using data collected at 293 K (Wei & Zheng, 2003).

(I)
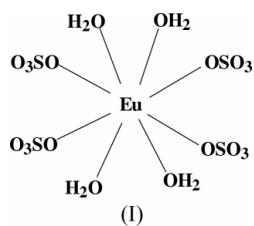
We report here the structure at 120 K. The similarity of the unit-cell dimensions and atomic coordinates at 293 and 120 K indicates that the same phase has been utilized in all of these studies. The aims of the present investigation are the determination of more precise metrical parameters and the determination of the extent of hydration. The structure (Table 1 and Fig. 1) indicates the presence of octacoordinate europium, with distorted square antiprismatic coordination by four water molecules, one O atom from a sulfate ion in the μ_2 bonding mode and three O atoms from three different anions in the μ_3 bonding mode. The triply bridging anions lie in general positions, while the doubly bridging anions lie on twofold rotation axes.
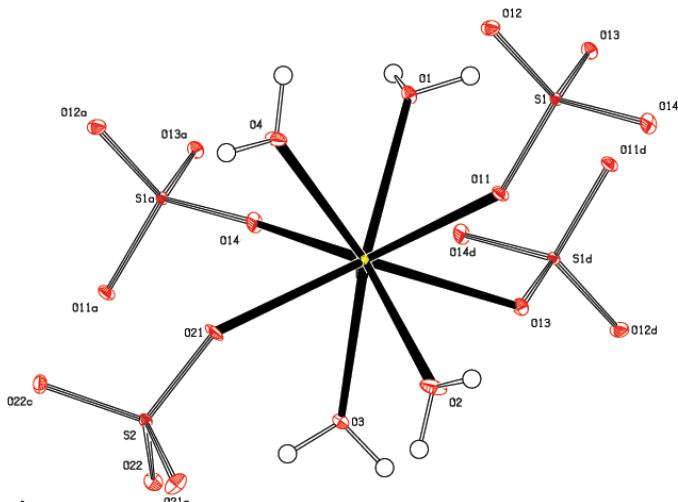
Figure 1
ORTEP diagram of (I), showing the coordination geometry around europium, with 50% probability ellipsoids. [Symmetry codes: (*a*) $-x$, *y*, $\frac{1}{2} - z$; (*b*) $\frac{1}{2} - x$, $\frac{1}{2} - y$, $1 - z$; (*c*) $\frac{1}{2} - x$, $\frac{3}{2} - y$, $1 - z$.]
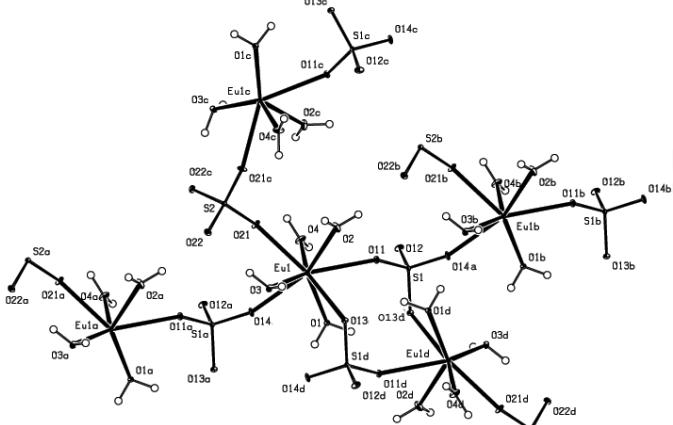

Figure 2
Crystal structure of (I), showing the sulfate coordination.

Figure 3
Packing diagram of (I), viewed along the *c* axis.

Compound (I) is, in fact, isostructural not only with yttrium(III) sulfate octahydrate (Held & Wickleder, 2003) but also with the analogous lanthanide sulfates $Ln_2(SO_4)_3 \cdot 8H_2O$, where Ln is Ce (Junk *et al.*, 1999), Pr (Ahmed Farag *et al.*, 1981), Nd (Bartl & Rodek, 1983), Sm (Podberezskaya & Borisov, 1976), Dy (Junk *et al.*, 1999), Er (Wickleder, 1999), Yb (Hiltunen & Niinistö, 1976) or Lu (Junk *et al.*, 1999). The coordination polymer in this structure is most readily envisaged as inversion-related pairs of chains comprising alternating cations and triply bridging anions, themselves generated by translation along the [010] direction, which are then linked into sheets by the doubly bridging anions. The coordination-polymer sheets are linked by hydrogen bonds (Table 2) into a continuous three-dimensional framework structure. As noted for the yttrium analogue (Held & Wickleder, 2003), one of the S—O bonds in the triply bridging anion is significantly longer than the others (Table 1).

Some of the lanthanides, such as europium, can also exhibit lower oxidation states in sulfate salts. Thus, for example, europium(II) sulfate has been shown to be anhydrous and to crystallize in space group *Pnma* (Mayer *et al.*, 1964).

Accordingly, the oxidation state of europium in (I) was further confirmed by bond valence sum calculations (Brown, 1992, 2002). A total valence of 3.016 for europium was obtained using the observed Eu—O bond lengths (Table 1) and a bond valence parameter of 2.036 Å for europium (Trzesowska *et al.*, 2004).

Experimental

The title compound was obtained during the attempted preparation of a complex between 2,5-diketopiperazine and europium sulfate, in which 2,5-diketopiperazine (0.228 g, 2 mmol) was heated with europium sulfate (0.736 g, 1 mmol) in water (30 ml). The latter was obtained by the action of sulfuric acid on europium oxide. The crystallization of europium sulfate from solution is facilitated in the presence of other ligands (Held & Wickleder, 2003; Wei & Zheng, 2003).

Crystal data

$Eu_2(SO_4)_3 \cdot 8H_2O$	$D_x = 3.002$ Mg m^{-3}
$M_r = 736.26$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 1869
$a = 13.5029$ (3) Å	reflections
$b = 6.7601$ (1) Å	$\theta = 3.1$–$27.5°$
$c = 18.2628$ (3) Å	$\mu = 8.12$ mm^{-1}
$\beta = 102.2610$ (13)°	$T = 120$ (2) K
$V = 1629.02$ (5) Å^3	Block, colourless
$Z = 4$	$0.10 \times 0.08 \times 0.06$ mm

Data collection

Bruker–Nonius KappaCCD diffractometer with FR591 rotating anode	1869 independent reflections
	1805 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.037$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	$\theta_{max} = 27.5°$
	$h = -17 \rightarrow 17$
$T_{min} = 0.477$, $T_{max} = 0.616$	$k = -8 \rightarrow 8$
11 784 measured reflections	$l = -22 \rightarrow 23$

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Refinement

Table 1
Selected interatomic distances(Å).

Eu1—O1	2.450 (3)	Eu1—O21	2.339 (2)
Eu1—O2	2.390 (2)	S1—O11^i	1.473 (2)
Eu1—O3	2.505 (2)	S1—O12	1.459 (3)
Eu1—O4	2.350 (3)	S1—O13	1.499 (2)
Eu1—O11	2.384 (2)	S1—O14^{ii}	1.463 (2)
Eu1—O13	2.461 (2)	S2—O21	1.470 (2)
Eu1—O14	2.336 (2)	S2—O22	1.478 (2)

Symmetry codes: (i) $\frac{1}{2} - x, \frac{3}{2} - y, 1 - z$; (ii) $\frac{1}{2} - x, \frac{1}{2} - y, 1 - z$.

Table 2
Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H11$\cdots$O3^i	0.84	2.08	2.910 (3)	170
O1—H12$\cdots$O13^{ii}	0.84	2.06	2.771 (3)	143
O2—H21$\cdots$O12^{iii}	0.84	1.87	2.704 (4)	171
O2—H22$\cdots$O22^{iv}	0.84	1.99	2.814 (3)	169
O3—H31$\cdots$O22	0.84	1.96	2.759 (3)	159
O3—H32$\cdots$O13^{iii}	0.84	2.16	2.994 (3)	170
O4—H41$\cdots$O12^v	0.84	1.93	2.748 (4)	163
O4—H42$\cdots$O22^{vi}	0.84	1.97	2.788 (4)	165

Symmetry codes: (i) $\frac{1}{2} - x, \frac{1}{2} - y, 1 - z$; (ii) $\frac{1}{2} - x, \frac{3}{2} - y, 1 - z$; (iii) $-x, 1 - y, 1 - z$; (iv) $x, 1 + y, z$; (v) $x, 1 - y, z - \frac{1}{2}$; (vi) $\frac{1}{2} + x, \frac{1}{2} + y, z$.

All H atoms were located in difference maps and then allowed to ride on their parent atoms, with O—H distances of 0.84 Å and with $U_{iso}(H) = 1.2U_{eq}(O)$.

Data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PRPKAPPA* (Ferguson, 1999).

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Dieuropium(III) trisulfate octahydrate: a redetermination at 120 K

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Dieuropium trisulfate octahydrate

Crystal data
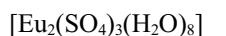
[Eu$_2$(SO$_4$)$_3$(H$_2$O)$_8$]
$M_r = 736.26$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 13.5029$ (3) Å
$b = 6.7601$ (1) Å
$c = 18.2628$ (3) Å
$\beta = 102.2610$ (13)°
$V = 1629.02$ (5) Å^3
$Z = 4$

$F(000) = 1400$
$D_x = 3.002$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1869 reflections
$\theta = 3.1$–27.5°
$\mu = 8.12$ mm^{-1}
$T = 120$ K
Plate, colourless
$0.10 \times 0.08 \times 0.06$ mm

Data collection

Bruker-Nonius 95mm CCD camera on κ-
  goniostat
  diffractometer
Radiation source: Bruker–Nonius FR591
  rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ & ω scans
Absorption correction: multi-scan
  (SADABS; Sheldrick, 2003)

$T_{min} = 0.477$, $T_{max} = 0.616$
11784 measured reflections
1869 independent reflections
1805 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
$h = -17 \rightarrow 17$
$k = -8 \rightarrow 8$
$l = -22 \rightarrow 23$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.019$
$wR(F^2) = 0.054$
$S = 1.26$
1869 reflections
114 parameters
0 restraints
Primary atom site location: structure-invariant
  direct methods

Secondary atom site location: difference Fourier
  map
Hydrogen site location: inferred from
  neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0218P)^2 + 6.1179P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.71$ e Å^{-3}
$\Delta\rho_{min} = -1.32$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Eu1	0.167587 (11)	0.47732 (2)	0.392539 (9)	0.00436 (8)
O1	0.34495 (19)	0.5163 (3)	0.45743 (14)	0.0075 (5)
O2	0.01431 (17)	0.6639 (4)	0.35904 (14)	0.0140 (5)
O3	0.04016 (16)	0.2634 (3)	0.43634 (13)	0.0083 (5)
O4	0.2592 (2)	0.5073 (4)	0.29716 (15)	0.0123 (5)
S1	0.21644 (6)	0.52970 (11)	0.58924 (5)	0.00457 (17)
O11	0.20077 (17)	0.8241 (3)	0.39758 (12)	0.0077 (5)
O12	0.16150 (18)	0.5354 (4)	0.64991 (14)	0.0097 (5)
O13	0.14441 (17)	0.5794 (3)	0.51715 (12)	0.0076 (4)
O14	0.24242 (17)	0.1670 (3)	0.41959 (13)	0.0103 (5)
S2	0.0000	0.17534 (16)	0.2500	0.0053 (2)
O21	0.08491 (17)	0.3008 (4)	0.28701 (13)	0.0106 (5)
O22	−0.03310 (19)	0.0515 (3)	0.30707 (14)	0.0089 (5)
H11	0.3814	0.4312	0.4838	0.009*
H12	0.3610	0.6166	0.4845	0.009*
H21	−0.0434	0.6132	0.3549	0.017*
H22	0.0076	0.7843	0.3477	0.017*
H31	0.0064	0.1908	0.4028	0.010*
H32	−0.0066	0.3184	0.4527	0.010*
H41	0.2369	0.4741	0.2524	0.015*
H42	0.3197	0.5429	0.3007	0.015*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Eu1	0.00432 (12)	0.00358 (11)	0.00492 (12)	−0.00052 (5)	0.00041 (8)	−0.00030 (5)
O1	0.0081 (12)	0.0060 (11)	0.0069 (12)	0.0006 (8)	−0.0017 (10)	−0.0003 (8)
O2	0.0059 (11)	0.0084 (12)	0.0258 (14)	−0.0008 (9)	−0.0012 (10)	0.0046 (10)
O3	0.0067 (11)	0.0085 (11)	0.0095 (11)	−0.0019 (9)	0.0008 (9)	−0.0017 (9)
O4	0.0083 (13)	0.0217 (13)	0.0073 (13)	−0.0068 (9)	0.0029 (11)	−0.0032 (9)
S1	0.0046 (4)	0.0032 (4)	0.0057 (4)	−0.0001 (3)	0.0007 (3)	0.0002 (3)
O11	0.0066 (11)	0.0053 (11)	0.0107 (12)	−0.0019 (9)	0.0008 (9)	−0.0003 (9)
O12	0.0072 (13)	0.0157 (12)	0.0066 (13)	−0.0018 (9)	0.0021 (10)	−0.0016 (9)
O13	0.0076 (11)	0.0081 (11)	0.0059 (11)	0.0001 (9)	−0.0010 (9)	0.0001 (9)
O14	0.0115 (12)	0.0029 (11)	0.0158 (12)	0.0019 (9)	0.0012 (9)	−0.0003 (9)
S2	0.0052 (5)	0.0049 (5)	0.0053 (5)	0.000	−0.0004 (4)	0.000
O21	0.0094 (11)	0.0139 (12)	0.0078 (11)	−0.0078 (9)	0.0006 (9)	−0.0041 (9)
O22	0.0104 (12)	0.0071 (11)	0.0091 (12)	−0.0008 (9)	0.0022 (10)	0.0023 (9)

Geometric parameters (Å, º)

Eu1—O1	2.450 (3)	O3—H31	0.84
Eu1—O2	2.390 (2)	O3—H32	0.84
Eu1—O3	2.505 (2)	O4—H41	0.84
Eu1—O4	2.350 (3)	O4—H42	0.84

Eu1—O11	2.384 (2)	S1—O11[i]	1.473 (2)
Eu1—O13	2.461 (2)	S1—O12	1.459 (3)
Eu1—O14	2.336 (2)	S1—O13	1.499 (2)
Eu1—O21	2.339 (2)	S1—O14[ii]	1.463 (2)
O1—H11	0.84	S2—O21	1.470 (2)
O1—H12	0.84	S2—O21[iii]	1.470 (2)
O2—H21	0.84	S2—O22	1.478 (2)
O2—H22	0.84	S2—O22[iii]	1.478 (2)
O14—Eu1—O21	79.93 (8)	O1—Eu1—S1	62.92 (6)
O14—Eu1—O4	87.81 (8)	O13—Eu1—S1	20.55 (5)
O21—Eu1—O4	70.58 (8)	O3—Eu1—S1	74.07 (5)
O14—Eu1—O11	143.67 (8)	Eu1—O1—H11	126.7
O21—Eu1—O11	125.90 (8)	Eu1—O1—H12	118.4
O4—Eu1—O11	79.62 (8)	H11—O1—H12	99.7
O14—Eu1—O2	147.20 (8)	Eu1—O2—H21	122.9
O21—Eu1—O2	79.78 (8)	Eu1—O2—H22	128.1
O4—Eu1—O2	109.18 (9)	H21—O2—H22	108.9
O11—Eu1—O2	68.68 (8)	Eu1—O3—H31	114.0
O14—Eu1—O1	70.11 (8)	Eu1—O3—H32	118.4
O21—Eu1—O1	134.41 (8)	H31—O3—H32	100.7
O4—Eu1—O1	74.62 (9)	Eu1—O4—H41	124.2
O11—Eu1—O1	73.70 (7)	Eu1—O4—H42	128.6
O2—Eu1—O1	140.48 (8)	H41—O4—H42	107.1
O14—Eu1—O13	100.98 (8)	O12—S1—O14[ii]	111.97 (14)
O21—Eu1—O13	140.61 (8)	O12—S1—O11[i]	110.40 (14)
O4—Eu1—O13	148.41 (8)	O14[ii]—S1—O11[i]	109.58 (13)
O11—Eu1—O13	75.44 (8)	O12—S1—O13	108.74 (14)
O2—Eu1—O13	79.37 (8)	O14[ii]—S1—O13	107.30 (14)
O1—Eu1—O13	79.97 (8)	O11[i]—S1—O13	108.75 (13)
O14—Eu1—O3	73.11 (8)	O12—S1—Eu1	139.49 (10)
O21—Eu1—O3	74.25 (8)	O14[ii]—S1—Eu1	77.87 (10)
O4—Eu1—O3	142.36 (8)	O11[i]—S1—Eu1	101.82 (9)
O11—Eu1—O3	133.73 (7)	S1[i]—O11—Eu1	142.72 (14)
O2—Eu1—O3	76.72 (8)	S1—O13—Eu1	124.27 (13)
O1—Eu1—O3	125.08 (8)	S1[ii]—O14—Eu1	161.88 (15)
O13—Eu1—O3	68.63 (8)	O21—S2—O21[iii]	109.6 (2)
O14—Eu1—S1	84.02 (6)	O21—S2—O22	108.99 (13)
O21—Eu1—S1	147.46 (6)	O21—S2—O22[iii]	109.13 (13)
O4—Eu1—S1	137.03 (7)	O22—S2—O22[iii]	111.0 (2)
O11—Eu1—S1	82.48 (5)	S2—O21—Eu1	149.58 (14)
O2—Eu1—S1	99.91 (6)		
O14—Eu1—S1—O12	−108.91 (17)	O2—Eu1—O11—S1[i]	−175.9 (2)
O21—Eu1—S1—O12	−48.3 (2)	O1—Eu1—O11—S1[i]	16.4 (2)
O4—Eu1—S1—O12	170.80 (18)	O13—Eu1—O11—S1[i]	100.0 (2)
O11—Eu1—S1—O12	104.91 (17)	O3—Eu1—O11—S1[i]	139.71 (19)
O2—Eu1—S1—O12	38.20 (17)	S1—Eu1—O11—S1[i]	80.3 (2)

O1—Eu1—S1—O12	−179.60 (17)	O12—S1—O13—Eu1	−155.83 (14)
O13—Eu1—S1—O12	36.7 (2)	O14ⁱⁱ—S1—O13—Eu1	−34.53 (19)
O3—Eu1—S1—O12	−34.81 (17)	O11ⁱ—S1—O13—Eu1	83.92 (17)
O14—Eu1—S1—O14ⁱⁱ	0.82 (15)	O14—Eu1—O13—S1	34.95 (17)
O21—Eu1—S1—O14ⁱⁱ	61.43 (15)	O21—Eu1—O13—S1	122.42 (16)
O4—Eu1—S1—O14ⁱⁱ	−79.46 (13)	O4—Eu1—O13—S1	−69.0 (2)
O11—Eu1—S1—O14ⁱⁱ	−145.36 (11)	O11—Eu1—O13—S1	−107.93 (16)
O2—Eu1—S1—O14ⁱⁱ	147.93 (11)	O2—Eu1—O13—S1	−178.45 (17)
O1—Eu1—S1—O14ⁱⁱ	−69.87 (11)	O1—Eu1—O13—S1	−32.33 (15)
O13—Eu1—S1—O14ⁱⁱ	146.39 (19)	O3—Eu1—O13—S1	101.77 (16)
O3—Eu1—S1—O14ⁱⁱ	74.93 (11)	O21—Eu1—O14—S1ⁱⁱ	31.0 (5)
O14—Eu1—S1—O11ⁱ	108.59 (11)	O4—Eu1—O14—S1ⁱⁱ	−39.7 (5)
O21—Eu1—S1—O11ⁱ	169.20 (14)	O11—Eu1—O14—S1ⁱⁱ	−108.8 (5)
O4—Eu1—S1—O11ⁱ	28.30 (14)	O2—Eu1—O14—S1ⁱⁱ	83.6 (5)
O11—Eu1—S1—O11ⁱ	−37.59 (14)	O1—Eu1—O14—S1ⁱⁱ	−114.1 (5)
O2—Eu1—S1—O11ⁱ	−104.30 (11)	O13—Eu1—O14—S1ⁱⁱ	170.9 (5)
O1—Eu1—S1—O11ⁱ	37.90 (11)	O3—Eu1—O14—S1ⁱⁱ	107.5 (5)
O13—Eu1—S1—O11ⁱ	−105.84 (19)	S1—Eu1—O14—S1ⁱⁱ	−177.4 (5)
O3—Eu1—S1—O11ⁱ	−177.30 (11)	O21ⁱⁱⁱ—S2—O21—Eu1	88.2 (3)
O14—Eu1—S1—O13	−145.56 (17)	O22—S2—O21—Eu1	−31.1 (3)
O21—Eu1—S1—O13	−84.96 (19)	O22ⁱⁱⁱ—S2—O21—Eu1	−152.5 (3)
O4—Eu1—S1—O13	134.15 (18)	O14—Eu1—O21—S2	88.9 (3)
O11—Eu1—S1—O13	68.25 (17)	O4—Eu1—O21—S2	−179.9 (3)
O2—Eu1—S1—O13	1.54 (17)	O11—Eu1—O21—S2	−119.3 (3)
O1—Eu1—S1—O13	143.74 (17)	O2—Eu1—O21—S2	−65.2 (3)
O3—Eu1—S1—O13	−71.46 (17)	O1—Eu1—O21—S2	137.7 (3)
O14—Eu1—O11—S1ⁱ	11.2 (3)	O13—Eu1—O21—S2	−6.2 (4)
O21—Eu1—O11—S1ⁱ	−117.1 (2)	O3—Eu1—O21—S2	13.7 (3)
O4—Eu1—O11—S1ⁱ	−60.4 (2)	S1—Eu1—O21—S2	27.2 (4)

Symmetry codes: (i) −*x*+1/2, −*y*+3/2, −*z*+1; (ii) −*x*+1/2, −*y*+1/2, −*z*+1; (iii) −*x*, *y*, −*z*+1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H11···O3ⁱⁱ	0.84	2.08	2.910 (3)	170
O1—H12···O13ⁱ	0.84	2.06	2.771 (3)	143
O2—H21···O12ⁱᵛ	0.84	1.87	2.704 (4)	171
O2—H22···O22ᵛ	0.84	1.99	2.814 (3)	169
O3—H31···O22	0.84	1.96	2.759 (3)	159
O3—H32···O13ⁱᵛ	0.84	2.16	2.994 (3)	170
O4—H41···O12ᵛⁱ	0.84	1.93	2.748 (4)	163
O4—H42···O22ᵛⁱⁱ	0.84	1.97	2.788 (4)	165

Symmetry codes: (i) −*x*+1/2, −*y*+3/2, −*z*+1; (ii) −*x*+1/2, −*y*+1/2, −*z*+1; (iv) −*x*, −*y*+1, −*z*+1; (v) *x*, *y*+1, *z*; (vi) *x*, −*y*+1, *z*−1/2; (vii) *x*+1/2, *y*+1/2, *z*.