Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Poly[μ_2-hydroxido-μ_4-sulfato-neodymium(III)]

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Received 2 July 2008; accepted 13 July 2008

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(Nd–O) = 0.004 Å; R factor = 0.023; wR factor = 0.057; data-to-parameter ratio = 10.1.

The title compound, $[Nd(OH)(SO_4)]_n$, was obtained hydrothermally from an aqueous solution of neodymium nitrate, 1,2-propanediamine and sulfuric acid. The structure features nonacoordinated neodymium with sulfate and hydroxide anions acting as bridging ligands. The OH group forms a weak O—H···O hydrogen bond with an O···O distance of 3.224 (5) Å.

Related literature

For related literature, see: Doran *et al.* (2002); Xu, Ding, Zhou & Liu (2006); Xu, Ding, Feng *et al.* (2006); Xu *et al.* (2007); Yuan *et al.* (2004); Zhang *et al.* (2004); Ding *et al.* (2006).

Experimental

Crystal data

$[Nd(OH)(SO_4)]$
M_r = 257.31
Monoclinic, $P2_1/n$
a = 4.4678 (9) Å
b = 12.432 (2) Å
c = 6.8575 (13) Å
β = 106.324 (3)°
V = 365.53 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 14.66 mm^{-1}
T = 293 (2) K
0.10 × 0.08 × 0.06 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.322, T_{max} = 0.473
(expected range = 0.282–0.415)
1837 measured reflections
675 independent reflections
669 reflections with $I > 2\sigma(I)$
R_{int} = 0.013

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.056
S = 1.24
675 reflections
67 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −2.27 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O5—H1···O1^{i}	0.83 (3)	2.43 (3)	3.224 (5)	160 (6)

Symmetry code: (i) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The authors thank Dr Zhang for help with the structural analysis.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BR2079).

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supplementary materials

Acta Cryst. (2008). E**64**, i49 [**doi:10.1107/S1600536808021818**]

Poly[μ_2-hydroxido-μ_4-sulfato-neodymium(III)]

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Comment

In the last few years, the synthesis of new three dimensional lanthanide sulfates have received great attention, due to their functional applications in catalysis, ion-exchange, and optical device (Zhang *et al.*,2004; Yuan *et al.*, 2004; Xu, Ding, Feng *et al.*, 2006; Xu, Ding, Zhou & Liu, 2006; Doran *et al.*, 2002, Xu *et al.*, 2007). In this work, we designed and synthesized the title compound, neodymium(3+) sulfate hydroxide, which features a three–dimensional framework constructed from NdO_9 polyhedra and SO_4 tetrahedra.

$Nd(SO_4)(OH)$ is isostructural with $La(SO_4)(OH)$ (Zhang *et al.*,2004) and $Eu(SO_4)(OH)$(Ding *et al.*,2006), the framework of title compound constructed from NdO_9 polyhedra and SO_4 tetrahedra. As show in Fig. 1, the asymmetric unit contains one Nd^{3+}, one SO_4^{2-} group and one hydroxide group. The Nd^{3+} is coordinated by six bridging sulfate ions, each S atom makes four S–O–Nd linkages by sharing the bridging O atoms. The coordination sphere of Nd is completed by three OH^- groups, which act as briding ligands between three Nd^3+^.

The O-H group is involved hydrogen bonding interactions with O1, O2 and O4, the distances of O—H···O are vary from 2.60 (2) to 2.90 (2) Å.

The Nd —O distances are between of 2.374 (4)– 2.800 (4)Å (Table 1)while the O—Nd—O angles are between 66.02 (13) and 141.55 (12)°. These bond distances and bond angles are in agreement with those found in the reported rare-earth compounds (Zhang *et al.*,2004; Ding *et al.*,2006). The bond distances of S—O and angles of O—S—O are unexceptional. Fig. 2 shows the three-dimensional arrangement in the unit cell, displaying the way the different Nd^{3+} are connected by bridging hydroxide and sulfates groups.

Experimental

Pink block crystals were synthesized hydrothermally from a mixture of $Nd(NO_3)_3.6H_2O$, 1,2-propane diamine, H_2SO_4 and water. In a typical synthesis, $Nd(NO_3)_3.6H_2O$ (0.6066 g) was dissolved in a mixture of 1,2-propane diamine (0.2205 g) and water (3.2 ml) followed by the addition of H_2SO_4 (98%) (0.3093 g) with constant stirring. Finally, the mixture was kept in a 25 ml Teflon-lined steel autoclave at 180 °C for 7 days. After the autoclave was slowly cooled to room temperature, Pink block crystals of the title compound were obtained.

Refinement

The H atom of water was located from difference map, while the distance of O—H was restrained as 0.85 (2) Å.

Figures

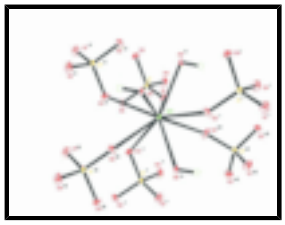

Fig. 1. The molecular structure for title compound. Displacement ellipsoids at the 50% probability level.

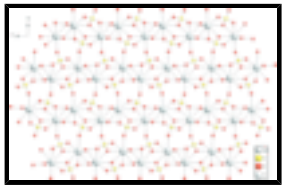

Fig. 2. The crystal packing in the unit cell of $Nd(SO_4)(OH)$.

Poly[μ_2-hydroxido-μ_4-sulfato-neodymium(III)]

Crystal data

$[Nd(OH)(SO_4)]$	$F_{000} = 468$
$M_r = 257.31$	$D_x = 4.676$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 150 reflections
$a = 4.4678$ (9) Å	$\theta = 2.3$–22.5º
$b = 12.432$ (2) Å	$\mu = 14.66$ mm^{-1}
$c = 6.8575$ (13) Å	$T = 293$ (2) K
$\beta = 106.324$ (3)º	Block, pink
$V = 365.53$ (12) Å^3	0.10 × 0.08 × 0.06 mm
$Z = 4$	

Data collection

Bruker APEXII CCD diffractometer	675 independent reflections
Radiation source: fine-focus sealed tube	669 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.013$
$T = 293$(2) K	$\theta_{max} = 25.5$º
ω scans	$\theta_{min} = 3.3$º
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	$h = -3 \rightarrow 5$
$T_{min} = 0.322$, $T_{max} = 0.473$	$k = -14 \rightarrow 15$
1837 measured reflections	$l = -7 \rightarrow 8$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.022$

$wR(F^2) = 0.056$

$S = 1.24$

675 reflections

67 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Hydrogen site location: difference Fourier map

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.035P)^2 + 0.7631P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.56$ e Å^{-3}

$\Delta\rho_{min} = -2.27$ e Å^{-3}

Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Nd1	0.14116 (6)	0.93569 (2)	0.80136 (4)	0.00655 (15)
S1	0.4852 (3)	0.85400 (10)	0.38900 (18)	0.0059 (3)
O1	0.3672 (9)	0.8343 (3)	0.5628 (6)	0.0136 (8)
O2	0.2485 (9)	0.9040 (3)	0.2196 (6)	0.0127 (8)
O3	0.7563 (9)	0.9295 (3)	0.4498 (6)	0.0105 (8)
O4	0.5923 (9)	0.7539 (3)	0.3200 (6)	0.0129 (8)
O5	0.3028 (9)	1.0847 (3)	1.0385 (6)	0.0081 (7)
H1	0.295 (14)	1.148 (2)	0.997 (9)	0.010*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Nd1	0.0075 (2)	0.0051 (2)	0.0073 (2)	0.00001 (8)	0.00245 (14)	−0.00073 (8)
S1	0.0073 (6)	0.0040 (6)	0.0068 (6)	0.0003 (4)	0.0026 (5)	−0.0001 (4)
O1	0.0172 (19)	0.0129 (19)	0.0136 (19)	0.0013 (16)	0.0093 (16)	0.0003 (15)
O2	0.0096 (18)	0.0133 (18)	0.0131 (18)	0.0026 (16)	−0.0002 (15)	0.0032 (15)
O3	0.0088 (19)	0.008 (2)	0.015 (2)	−0.0023 (13)	0.0036 (16)	−0.0004 (13)
O4	0.0196 (19)	0.0066 (19)	0.015 (2)	0.0037 (15)	0.0092 (17)	−0.0002 (14)
O5	0.0080 (18)	0.0034 (16)	0.0122 (18)	0.0005 (14)	0.0016 (14)	0.0027 (14)

Geometric parameters (Å, °)

Nd1—O4^{i}	2.374 (4)	S1—O1	1.453 (4)
Nd1—O5^{ii}	2.431 (4)	S1—O4	1.459 (4)
Nd1—O5	2.437 (4)	S1—O2	1.470 (4)
Nd1—O1	2.492 (4)	S1—O3	1.496 (4)
Nd1—O3^{iii}	2.535 (4)	O2—Nd1^{vi}	2.624 (4)
Nd1—O5^{iv}	2.536 (4)	O2—Nd1^{viii}	2.800 (4)
Nd1—O3^{v}	2.538 (4)	O3—Nd1^{iii}	2.535 (4)
Nd1—O2^{vi}	2.624 (4)	O3—Nd1^{ix}	2.538 (4)
Nd1—O2^{vii}	2.800 (4)	O4—Nd1^{x}	2.374 (4)
Nd1—Nd1^{iv}	3.6744 (7)	O5—Nd1^{ii}	2.431 (4)
Nd1—Nd1^{ii}	3.9178 (7)	O5—Nd1^{iv}	2.536 (4)
O4^{i}—Nd1—O5^{ii}	88.26 (13)	O4^{i}—Nd1—Nd1^{iv}	109.65 (10)
O4^{i}—Nd1—O5	137.19 (13)	O5^{ii}—Nd1—Nd1^{iv}	103.34 (9)
O5^{ii}—Nd1—O5	72.81 (14)	O5—Nd1—Nd1^{iv}	43.41 (9)
O4^{i}—Nd1—O1	66.02 (13)	O1—Nd1—Nd1^{iv}	173.45 (9)
O5^{ii}—Nd1—O1	72.10 (13)	O3^{iii}—Nd1—Nd1^{iv}	112.42 (8)
O5—Nd1—O1	136.35 (13)	O5^{iv}—Nd1—Nd1^{iv}	41.33 (8)
O4^{i}—Nd1—O3^{iii}	136.85 (12)	O3^{v}—Nd1—Nd1^{iv}	115.79 (9)
O5^{ii}—Nd1—O3^{iii}	91.10 (13)	O2^{vi}—Nd1—Nd1^{iv}	49.41 (8)
O5—Nd1—O3^{iii}	82.80 (13)	O2^{vii}—Nd1—Nd1^{iv}	45.37 (8)
O1—Nd1—O3^{iii}	72.80 (12)	O4^{i}—Nd1—Nd1^{ii}	115.94 (9)
O4^{i}—Nd1—O5^{iv}	77.43 (13)	O5^{ii}—Nd1—Nd1^{ii}	36.45 (9)
O5^{ii}—Nd1—O5^{iv}	128.19 (16)	O5—Nd1—Nd1^{ii}	36.35 (9)
O5—Nd1—O5^{iv}	84.74 (13)	O1—Nd1—Nd1^{ii}	105.03 (9)
O1—Nd1—O5^{iv}	138.09 (13)	O3^{iii}—Nd1—Nd1^{ii}	86.21 (9)
O3^{iii}—Nd1—O5^{iv}	132.32 (12)	O5^{iv}—Nd1—Nd1^{ii}	109.06 (9)
O4^{i}—Nd1—O3^{v}	88.46 (12)	O3^{v}—Nd1—Nd1^{ii}	151.19 (8)
O5^{ii}—Nd1—O3^{v}	139.42 (13)	O2^{vi}—Nd1—Nd1^{ii}	97.41 (9)
O5—Nd1—O3^{v}	130.63 (11)	O2^{vii}—Nd1—Nd1^{ii}	58.27 (8)
O1—Nd1—O3^{v}	69.68 (13)	Nd1^{iv}—Nd1—Nd1^{ii}	72.017 (16)
O3^{iii}—Nd1—O3^{v}	65.06 (14)	O1—S1—O4	110.5 (2)
O5^{iv}—Nd1—O3^{v}	90.27 (13)	O1—S1—O2	111.9 (2)
O4^{i}—Nd1—O2^{vi}	133.45 (13)	O4—S1—O2	109.4 (2)
O5^{ii}—Nd1—O2^{vi}	133.02 (12)	O1—S1—O3	109.2 (2)
O5—Nd1—O2^{vi}	61.74 (12)	O4—S1—O3	108.2 (2)
O1—Nd1—O2^{vi}	137.14 (12)	O2—S1—O3	107.5 (2)
O3^{iii}—Nd1—O2^{vi}	72.81 (13)	S1—O1—Nd1	139.5 (2)
O5^{iv}—Nd1—O2^{vi}	60.80 (12)	S1—O2—Nd1^{vi}	133.1 (2)

O3^{v}—Nd1—O2^{vi}	73.07 (12)	S1—O2—Nd1^{viii}	138.3 (2)
O4^{i}—Nd1—O2^{vii}	78.28 (12)	Nd1^{vi}—O2—Nd1^{viii}	85.22 (11)
O5^{ii}—Nd1—O2^{vii}	70.32 (12)	S1—O3—Nd1^{iii}	120.8 (2)
O5—Nd1—O2^{vii}	59.36 (12)	S1—O3—Nd1^{ix}	124.3 (2)
O1—Nd1—O2^{vii}	128.08 (12)	Nd1^{iii}—O3—Nd1^{ix}	114.94 (14)
O3^{iii}—Nd1—O2^{vii}	141.03 (12)	S1—O4—Nd1^{x}	155.2 (3)
O5^{iv}—Nd1—O2^{vii}	58.11 (12)	Nd1^{ii}—O5—Nd1	107.19 (14)
O3^{v}—Nd1—O2^{vii}	147.55 (12)	Nd1^{ii}—O5—Nd1^{iv}	128.19 (16)
O2^{vi}—Nd1—O2^{vii}	94.78 (11)	Nd1—O5—Nd1^{iv}	95.26 (12)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H1···O1^{xi}	0.83 (3)	2.43 (3)	3.224 (5)	160 (6)

Symmetry codes: (xi) −*x*+1/2, *y*+1/2, −*z*+3/2.

Fig. 1

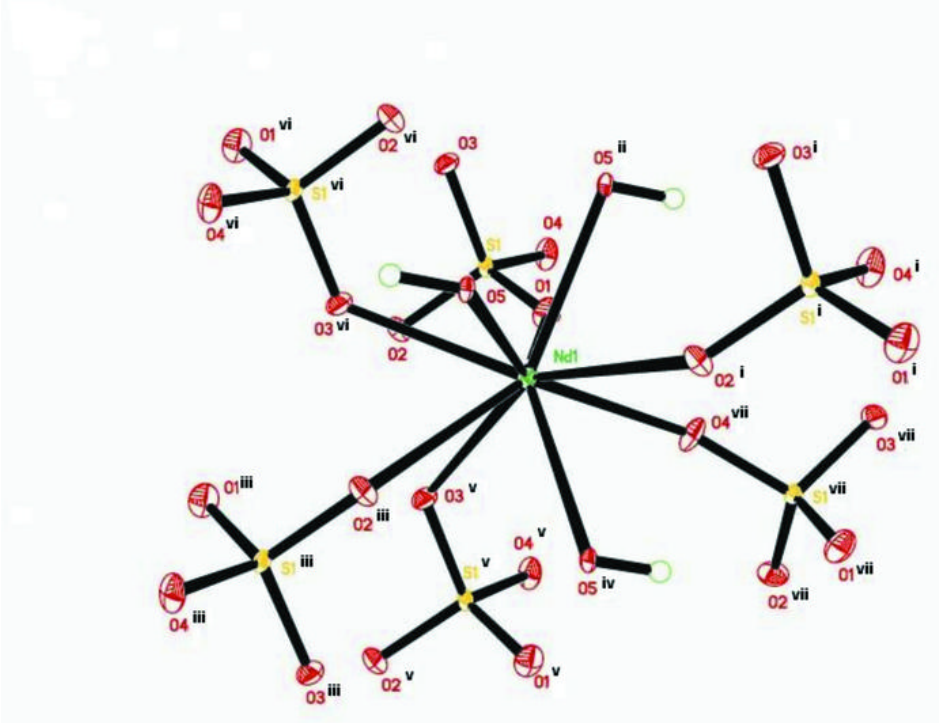
Nd1
O1 vi
O2 vi
Si1 vi
O4 vi
O3 vi
O2
O3
Si1
O4
O1
O5
O5 ii
O3 i
O4 i
Si1 i
O1 i
O2 i
O4 vii
O3 vii
Si1 vii
O1 vii
O2 vii
O1 iii
O2 iii
Si1 iii
O4 iii
O3 iii
O3 v
O4 v
Si1 v
O2 v
O1 v
O5 iv

Fig. 2