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Crystal structure of tris(ethylenediammonium) hexasulfatopraseodymium(III) hexahydrate

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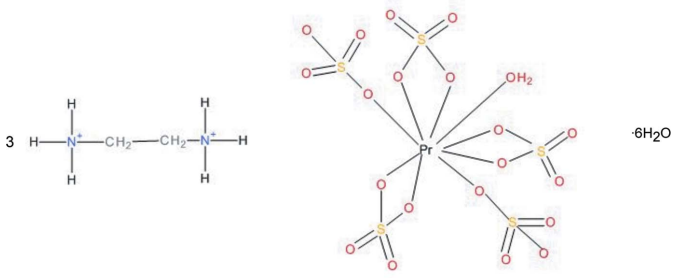
In the title salt, $(C_2H_{10}N_2)_3[Pr_2(SO_4)_6]\cdot 6H_2O$, the Pr^{III} cation is surrounded ninefold by five sulfate groups (two monodentate and three chelating) and by one water molecule [range of Pr—O bond lengths 2.383 (3) to 2.582 (3) Å]. The $[Pr(SO_4)_5(H_2O)]$ groups are arranged in sheets parallel to (010). Two crystal water molecules and two ethylenediammonium cations (one with point group symmetry $\overline{1}$) connect the sheets *via* O—H···O and N—H···O hydrogen bonds from weak up to medium strength into a three-dimensional framework structure.

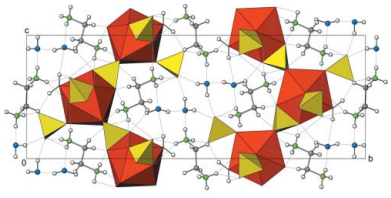
1. Chemical context

In the course of a systematic search for new 'double salts' of simple secondary amines and mono- or divalent cations of various inorganic acids, the structures of $(C_2H_{10}N_2)[Li_2(SO_4)_2]$ and $(C_2H_8N)[Cu(HSO_4)(SO_4)(H_2O)_4]$ have been described previously (Held, 2003, 2014). In continuation of these studies, lithium was replaced by trivalent praseodymium, yielding crystals of the title compound with composition $(C_2H_{10}N_2)_3[Pr_2(SO_4)_6]\cdot 6H_2O$.



2. Structural commentary

The asymmetric unit of the title compound contains three $(SO_4)^{2-}$ anions, one and a half $[NH_2(CH_3)]^{2+}$ cations (the other half being generated by inversion symmetry), one Pr^{3+} cation as well as three water molecules (Fig. 1). The Pr^{3+} cation is surrounded by nine O atoms from five sulfate groups, two of which are monodentately bonding and three chelating, and of one water molecule. The averaged Pr—O distance in the resulting distorted monocapped square-antiprism, $[Pr(SO_4)_5(H_2O)]$, is 2.52 (7) Å. Praseodymium reaches an overall bond-valence sum (Brown & Altermatt, 1985) of 3.23 valence units. The S—O distances are nearly equal [average distance 1.479 (13) Å], however, the O—S—O angles vary [average bond angle 109.48 (2.05)°] clearly. One sulfate group (S2) interconnects two $[PrO_9]$ polyhedra *via* two common edges parallel to [001], while another sulfate group (S3)



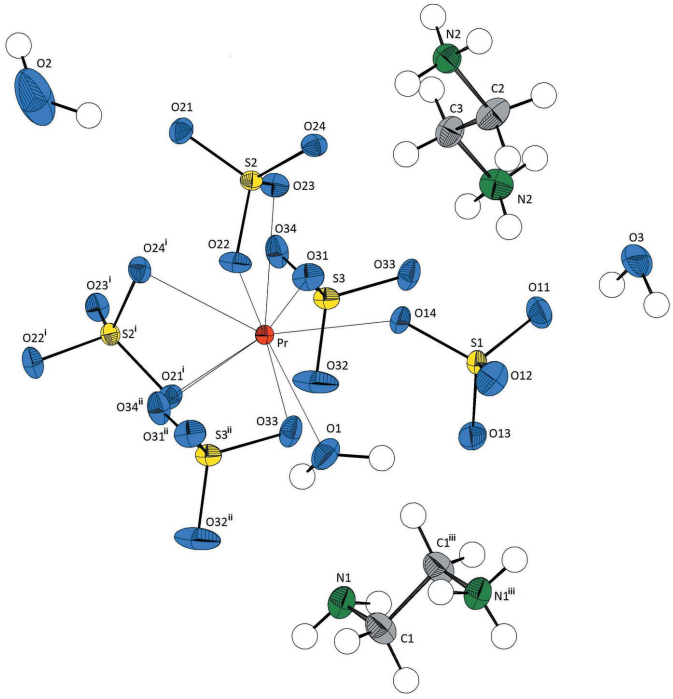


Figure 1
The molecular entities in the structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $x+1, y, z$; (iii) $-x, -y, -z+1$].

connects *via* a common edge and a common vertex parallel to [100], leading to the formation of sheets parallel to (010).

3. Supramolecular features

Hydrogen bonds of medium strength involving water molecules as donor groups and O atoms of the sulfate anions as acceptor groups interconnect neighbouring $[Pr(SO_4)_5(H_2O)]$ units. Together with relatively weaker N—H···O hydrogen bonds of the ammonium groups atoms to sulfate anions, a three-dimensional framework is formed (Table 1, Fig. 2).

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H11···O32	0.72 (8)	2.53 (8)	2.974 (6)	121 (7)
O1—H12···O13	0.78 (6)	1.92 (6)	2.674 (5)	162 (6)
O2—H21···O3	1.00 (12)	1.89 (12)	2.858 (7)	163 (9)
O2—H22···O21^{i}	0.77 (8)	2.29 (8)	2.905 (6)	137 (7)
O3—H31···O11^{ii}	0.87 (7)	1.95 (8)	2.795 (5)	165 (7)
O3—H32···O12^{iii}	0.80 (8)	2.00 (8)	2.766 (5)	162 (8)
N1—H1*A*···O33	0.87 (8)	2.48 (8)	3.291 (5)	155 (6)
N1—H1*B*···O3	0.88 (7)	1.92 (7)	2.758 (6)	158 (6)
N1—H1*C*···O13^{iv}	0.99 (9)	1.85 (9)	2.841 (6)	176 (7)
N2—H2*A*···O24	0.76 (7)	2.21 (7)	2.976 (5)	177 (7)
N2—H2*B*···O22^{v}	0.83 (8)	2.17 (8)	2.967 (6)	162 (7)
N2—H2*C*···O34^{vi}	0.94 (7)	2.20 (6)	3.020 (5)	146 (5)
N3—H3*A*···O2^{vii}	0.85 (7)	2.12 (7)	2.901 (8)	153 (6)
N3—H3*B*···O11	0.90 (7)	1.95 (8)	2.847 (6)	175 (6)
N3—H3*C*···O33	0.87 (7)	2.20 (7)	3.066 (5)	173 (6)

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

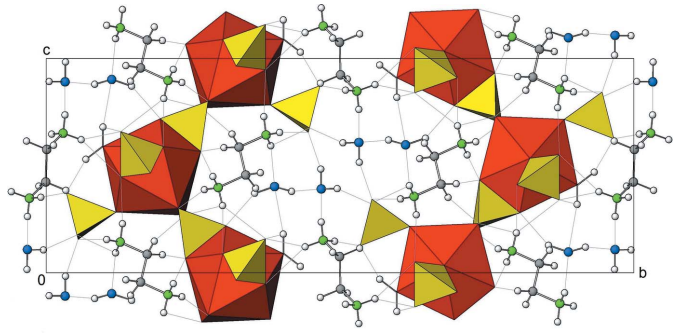


Figure 2
(100)-projection of the crystal structure of the title compound. Hydrogen bonds are shown as light-grey dashed lines. Colour scheme: (SO_4) tetrahedra (yellow), monocapped antiprism [PrO_9] (red), O (blue), N (green), C (grey), H (white).

4. Synthesis and crystallization

The title compound was obtained by reaction of an aqueous solution of praseodymium(III) sulfate with ethylenediamine and sulfuric acid (18 mol/l) in a stoichiometric ratio 1:1:2. The title compound crystallized by slow evaporation of the solvent at room temperature in form of light-green crystals with dimensions up to 3 mm within a few weeks.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were clearly

Table 2
Experimental details.

Crystal data	
Chemical formula	$(C_2H_{10}N_2)_3[Pr_2(SO_4)_6]\cdot 6H_2O$
M_r	1152.70
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	295
a, b, c (Å)	6.6174 (8), 26.668 (4), 10.0264 (13)
β (°)	104.446 (15)
V (Å^3)	1713.4 (4)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	3.29
Crystal size (mm)	0.22 × 0.21 × 0.20
Data collection	
Diffractometer	Stoe *IPDS*-II
Absorption correction	Multi-scan (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2002)
T_{min}, T_{max}	0.491, 0.620
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	14346, 3922, 3091
R_{int}	0.044
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.662
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.028, 0.069, 0.97
No. of reflections	3923
No. of parameters	311
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.72, −1.08

Computer programs: *X-AREA* (Stoe & Cie, 2002), *SIR92* (Altomare *et al.*, 1993), *SHELXL97* (Sheldrick, 2008), *ATOMS* (Dowty, 2002) and *publCIF* (Westrip, 2010).

discernible from difference Fourier maps. Methylene H atoms were refined with a riding-model constraint, using a C—H distance of 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C). Ammonium and water H atoms were refined freely.

Acknowledgements

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supporting information

Acta Cryst. (2014). E**70**, 235-237 [doi:10.1107/S1600536814020704]

Crystal structure of tris(ethylenediammonium) hexasulfatopraseodymium(III) hexahydrate

Peter Held

Computing details

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA* (Stoe & Cie, 2002); data reduction: *X-AREA* (Stoe & Cie, 2002); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ATOMS* (Dowty, 2002); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Tris(ethylenediammonium) hexasulfatopraseodymium(III) hexahydrate

Crystal data

$(C_2H_{10}N_2)_3[Pr_2(SO_4)_6]\cdot 6H_2O$
$M_r = 1152.70$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 6.6174\ (8)$ Å
$b = 26.668\ (4)$ Å
$c = 10.0264\ (13)$ Å
$\beta = 104.446\ (15)°$
$V = 1713.4\ (4)$ Å^3
$Z = 2$
$F(000) = 1148$
$D_x = 2.234$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 20.0–24.3°$
$\mu = 3.29$ mm^{-1}
$T = 295$ K
Parallelepiped, light-green
0.22 × 0.21 × 0.20 mm

Data collection

Stoe IPDS-II diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2002)
$T_{min} = 0.491$, $T_{max} = 0.620$
14346 measured reflections
3922 independent reflections
3091 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 28.1°$, $\theta_{min} = 2.6°$
$h = -8\rightarrow 8$
$k = -34\rightarrow 35$
$l = -13\rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.069$
$S = 0.97$
3923 reflections
311 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
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.72$ e Å^{-3}
$\Delta\rho_{min} = -1.08$ e Å^{-3}

Extinction correction: *SHELXL*,
$Fc^{*}=kFc[1+0.001xFc^{2}\lambda^{3}/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0150 (6)

Special details

Experimental. A suitable single-crystal was carefully selected under a polarizing microscope and mounted in a glass capillary. The scattering intensities were collected on an imaging plate diffractometer (*IPDS* II, Stoe & Cie) equipped with a fine focus sealed tube X-ray source (Mo Kα, λ = 0.71073 Å) operating at 50 kV and 30 mA. Intensity data for the title compound were collected at room temperature by ω-scans in 180 frames ($0 < \omega < 180°$; $\varphi = 0°$ and 90°, $\Delta\omega = 2°$, exposure time of 10 min) in the 2Θ range 2.29 to 59.53°. Structure solution and refinement were carried out using the programs *SIR97* (Altomare *et al.*, 1999) and *SHELXL97* (Sheldrick, 2008). The last cycles of refinement included atomic positions and anisotropic parameters for all atoms. The final difference maps were free of any chemically significant features. The refinement was based on F^2 for ALL reflections.

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}
Pr	0.54622 (3)	0.177147 (7)	0.465046 (19)	0.01075 (8)
S1	0.40656 (17)	0.06427 (3)	0.24648 (10)	0.0167 (2)
S2	0.52984 (15)	0.25100 (3)	0.21431 (9)	0.01307 (18)
S3	0.03251 (16)	0.15704 (4)	0.54283 (9)	0.0158 (2)
O11	0.1979 (5)	0.05421 (12)	0.1565 (3)	0.0283 (7)
O12	0.5682 (6)	0.05230 (12)	0.1764 (3)	0.0294 (7)
O13	0.4331 (5)	0.03414 (11)	0.3740 (3)	0.0269 (7)
O14	0.4203 (5)	0.11866 (10)	0.2836 (3)	0.0259 (7)
O21	0.5603 (5)	0.30594 (11)	0.2203 (3)	0.0205 (6)
O22	0.7151 (5)	0.22494 (11)	0.3007 (3)	0.0205 (6)
O23	0.3524 (5)	0.23557 (10)	0.2694 (3)	0.0183 (6)
O24	0.4972 (5)	0.23684 (10)	0.0667 (3)	0.0177 (6)
O31	0.1767 (5)	0.17700 (11)	0.4653 (3)	0.0222 (6)
O32	0.1426 (6)	0.12685 (14)	0.6597 (4)	0.0382 (9)
O33	−0.1327 (5)	0.12693 (11)	0.4480 (3)	0.0235 (6)
O34	−0.0807 (5)	0.19921 (12)	0.5890 (3)	0.0227 (6)
O1	0.5360 (6)	0.09619 (12)	0.5899 (4)	0.0298 (8)
H11	0.505 (12)	0.097 (3)	0.654 (8)	0.06 (2)*
H12	0.501 (9)	0.074 (2)	0.539 (6)	0.030 (15)*
O2	0.5314 (10)	0.1112 (3)	0.9028 (7)	0.100 (3)
H21	0.438 (17)	0.082 (4)	0.883 (10)	0.12 (4)*
H22	0.495 (12)	0.138 (3)	0.877 (8)	0.06 (2)*
O3	0.2361 (7)	0.03214 (16)	0.8914 (4)	0.0380 (9)
H31	0.220 (11)	0.033 (3)	0.975 (8)	0.056 (19)*
H32	0.310 (13)	0.009 (3)	0.888 (8)	0.07 (3)*

N1	−0.0784 (7)	0.02857 (16)	0.6520 (5)	0.0283 (8)
H1A	−0.090 (11)	0.060 (3)	0.623 (7)	0.06 (2)*
H1B	−0.005 (11)	0.032 (3)	0.738 (7)	0.053 (19)*
H1C	−0.204 (14)	0.008 (3)	0.646 (9)	0.08 (3)*
C1	0.0524 (8)	0.0008 (2)	0.5757 (5)	0.0300 (10)
H1D	0.082 (10)	−0.034 (3)	0.615 (6)	0.048 (17)*
H1E	0.173 (11)	0.014 (2)	0.595 (6)	0.045 (18)*
N2	0.0775 (8)	0.20727 (16)	−0.1020 (4)	0.0257 (8)
H2A	0.183 (11)	0.216 (2)	−0.057 (7)	0.040 (18)*
H2B	−0.011 (12)	0.230 (3)	−0.113 (7)	0.06 (2)*
H2C	0.086 (10)	0.199 (2)	−0.192 (7)	0.042 (16)*
C2	0.0133 (9)	0.16060 (18)	−0.0433 (5)	0.0278 (10)
H2D	0.115 (10)	0.136 (2)	−0.031 (6)	0.044 (17)*
H2E	−0.095 (13)	0.144 (3)	−0.107 (8)	0.08 (3)*
N3	−0.1249 (8)	0.12597 (16)	0.1435 (5)	0.0279 (9)
H3A	−0.245 (11)	0.119 (2)	0.094 (6)	0.039 (17)*
H3B	−0.023 (12)	0.103 (3)	0.153 (7)	0.06 (2)*
H3C	−0.126 (11)	0.129 (3)	0.230 (8)	0.05 (2)*
C3	−0.0508 (8)	0.17187 (17)	0.0867 (5)	0.0257 (9)
H3D	0.054 (10)	0.184 (2)	0.148 (6)	0.031 (15)*
H3E	−0.145 (9)	0.197 (2)	0.085 (6)	0.035 (15)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pr	0.01069 (13)	0.01111 (11)	0.01082 (11)	0.00001 (7)	0.00336 (7)	−0.00053 (8)
S1	0.0203 (6)	0.0136 (4)	0.0153 (4)	−0.0014 (3)	0.0026 (3)	−0.0027 (3)
S2	0.0144 (5)	0.0144 (4)	0.0107 (4)	0.0007 (3)	0.0038 (3)	0.0022 (3)
S3	0.0113 (5)	0.0182 (4)	0.0184 (4)	0.0002 (3)	0.0046 (3)	0.0041 (4)
O11	0.0219 (18)	0.0306 (17)	0.0283 (16)	−0.0023 (13)	−0.0014 (13)	−0.0069 (13)
O12	0.032 (2)	0.0285 (17)	0.0332 (17)	0.0017 (13)	0.0178 (14)	−0.0027 (13)
O13	0.037 (2)	0.0216 (15)	0.0219 (15)	−0.0055 (13)	0.0063 (13)	0.0041 (12)
O14	0.038 (2)	0.0132 (14)	0.0225 (15)	−0.0009 (12)	0.0005 (13)	−0.0048 (11)
O21	0.0296 (18)	0.0156 (13)	0.0170 (13)	−0.0024 (11)	0.0070 (12)	−0.0003 (10)
O22	0.0148 (16)	0.0265 (15)	0.0203 (14)	0.0032 (11)	0.0045 (11)	0.0095 (11)
O23	0.0148 (16)	0.0227 (14)	0.0192 (13)	−0.0006 (11)	0.0075 (11)	0.0064 (11)
O24	0.0223 (16)	0.0190 (14)	0.0128 (13)	0.0025 (11)	0.0064 (11)	0.0012 (10)
O31	0.0144 (16)	0.0258 (15)	0.0290 (15)	−0.0014 (11)	0.0101 (11)	0.0065 (12)
O32	0.0218 (19)	0.049 (2)	0.042 (2)	0.0036 (15)	0.0032 (15)	0.0301 (17)
O33	0.0167 (17)	0.0166 (14)	0.0376 (17)	−0.0033 (11)	0.0076 (12)	−0.0072 (12)
O34	0.0159 (17)	0.0282 (16)	0.0243 (15)	−0.0017 (12)	0.0057 (12)	−0.0115 (12)
O1	0.050 (2)	0.0174 (16)	0.0246 (17)	−0.0035 (14)	0.0150 (16)	−0.0012 (14)
O2	0.067 (4)	0.106 (5)	0.101 (5)	−0.043 (4)	−0.026 (3)	0.074 (4)
O3	0.049 (3)	0.039 (2)	0.0219 (17)	0.0071 (18)	0.0004 (15)	−0.0071 (15)
N1	0.030 (2)	0.023 (2)	0.033 (2)	−0.0027 (16)	0.0083 (17)	−0.0027 (17)
C1	0.023 (3)	0.037 (3)	0.028 (2)	0.001 (2)	0.0030 (19)	−0.004 (2)
N2	0.026 (3)	0.025 (2)	0.026 (2)	−0.0076 (17)	0.0056 (17)	0.0038 (16)
C2	0.036 (3)	0.023 (2)	0.030 (2)	0.0009 (19)	0.020 (2)	0.0038 (18)

N3	0.026 (3)	0.033 (2)	0.028 (2)	−0.0022 (17)	0.0126 (18)	0.0050 (17)
C3	0.028 (3)	0.022 (2)	0.030 (2)	−0.0035 (18)	0.0127 (19)	0.0000 (18)

Geometric parameters (Å, º)

Pr—O14	2.383 (3)	O34—Pr^{iv}	2.541 (3)
Pr—O31	2.446 (3)	O1—H11	0.72 (8)
Pr—O1	2.505 (3)	O1—H12	0.78 (6)
Pr—O34^{i}	2.541 (3)	O2—H21	1.00 (12)
Pr—O22	2.551 (3)	O2—H22	0.77 (8)
Pr—O33^{i}	2.553 (3)	O3—H31	0.87 (7)
Pr—O24^{ii}	2.564 (3)	O3—H32	0.80 (8)
Pr—O21^{ii}	2.577 (3)	N1—C1	1.487 (6)
Pr—O23	2.582 (3)	N1—H1A	0.87 (8)
Pr—S3^{i}	3.1621 (11)	N1—H1B	0.88 (7)
Pr—S2^{ii}	3.1725 (9)	N1—H1C	0.99 (9)
Pr—S2	3.1741 (9)	C1—C1^{v}	1.504 (9)
S1—O12	1.454 (3)	C1—H1D	1.01 (7)
S1—O11	1.473 (3)	C1—H1E	0.84 (7)
S1—O13	1.483 (3)	N2—C2	1.483 (6)
S1—O14	1.495 (3)	N2—H2A	0.76 (7)
S2—O23	1.475 (3)	N2—H2B	0.83 (8)
S2—O21	1.478 (3)	N2—H2C	0.94 (7)
S2—O22	1.485 (3)	C2—C3	1.499 (7)
S2—O24	1.490 (3)	C2—H2D	0.92 (7)
S3—O32	1.458 (3)	C2—H2E	0.94 (8)
S3—O31	1.472 (3)	N3—C3	1.484 (6)
S3—O34	1.488 (3)	N3—H3A	0.85 (7)
S3—O33	1.492 (3)	N3—H3B	0.90 (7)
O21—Pr^{iii}	2.577 (3)	N3—H3C	0.87 (7)
O24—Pr^{iii}	2.564 (3)	C3—H3D	0.87 (6)
O33—Pr^{iv}	2.553 (3)	C3—H3E	0.92 (6)
O14—Pr—O31	80.82 (11)	O31—S3—O33	109.05 (18)
O14—Pr—O1	76.67 (11)	O34—S3—O33	105.04 (17)
O31—Pr—O1	81.17 (12)	S1—O14—Pr	144.42 (18)
O14—Pr—O34^{i}	129.57 (10)	S2—O21—Pr^{iii}	99.34 (13)
O31—Pr—O34^{i}	148.16 (10)	S2—O22—Pr	100.34 (14)
O1—Pr—O34^{i}	95.70 (12)	S2—O23—Pr	99.30 (14)
O14—Pr—O22	87.71 (10)	S2—O24—Pr^{iii}	99.60 (14)
O31—Pr—O22	126.92 (9)	S3—O31—Pr	141.33 (18)
O1—Pr—O22	145.50 (11)	S3—O33—Pr^{iv}	99.49 (14)
O34^{i}—Pr—O22	70.82 (9)	S3—O34—Pr^{iv}	100.13 (14)
O14—Pr—O33^{i}	75.16 (10)	Pr—O1—H11	117 (6)
O31—Pr—O33^{i}	148.01 (9)	Pr—O1—H12	112 (4)
O1—Pr—O33^{i}	73.03 (12)	H11—O1—H12	121 (7)
O34^{i}—Pr—O33^{i}	55.31 (9)	H21—O2—H22	122 (8)
O22—Pr—O33^{i}	73.30 (10)	H31—O3—H32	107 (7)

O14—Pr—O24^{ii}	146.56 (10)	C1—N1—H1A	108 (5)
O31—Pr—O24^{ii}	77.02 (10)	C1—N1—H1B	108 (5)
O1—Pr—O24^{ii}	123.41 (11)	H1A—N1—H1B	102 (6)
O34^{i}—Pr—O24^{ii}	78.58 (9)	C1—N1—H1C	106 (5)
O22—Pr—O24^{ii}	85.94 (9)	H1A—N1—H1C	120 (7)
O33^{i}—Pr—O24^{ii}	133.31 (9)	H1B—N1—H1C	111 (6)
O14—Pr—O21^{ii}	142.34 (10)	N1—C1—C1^{v}	110.7 (5)
O31—Pr—O21^{ii}	77.75 (10)	N1—C1—H1D	110 (4)
O1—Pr—O21^{ii}	69.73 (11)	C1^{v}—C1—H1D	111 (4)
O34^{i}—Pr—O21^{ii}	71.56 (10)	N1—C1—H1E	109 (4)
O22—Pr—O21^{ii}	129.86 (10)	C1^{v}—C1—H1E	114 (4)
O33^{i}—Pr—O21^{ii}	109.50 (10)	H1D—C1—H1E	102 (5)
O24^{ii}—Pr—O21^{ii}	54.92 (8)	C2—N2—H2A	109 (5)
O14—Pr—O23	78.58 (10)	C2—N2—H2B	114 (5)
O31—Pr—O23	72.22 (9)	H2A—N2—H2B	112 (7)
O1—Pr—O23	146.05 (12)	C2—N2—H2C	106 (4)
O34^{i}—Pr—O23	118.03 (10)	H2A—N2—H2C	112 (6)
O22—Pr—O23	54.71 (9)	H2B—N2—H2C	104 (6)
O33^{i}—Pr—O23	121.97 (10)	N2—C2—C3	110.4 (4)
O24^{ii}—Pr—O23	70.99 (9)	N2—C2—H2D	112 (4)
O21^{ii}—Pr—O23	122.48 (9)	C3—C2—H2D	112 (4)
O12—S1—O11	110.7 (2)	N2—C2—H2E	112 (5)
O12—S1—O13	110.8 (2)	C3—C2—H2E	110 (5)
O11—S1—O13	108.69 (19)	H2D—C2—H2E	100 (6)
O12—S1—O14	109.09 (19)	C3—N3—H3A	108 (4)
O11—S1—O14	108.60 (19)	C3—N3—H3B	107 (5)
O13—S1—O14	108.92 (17)	H3A—N3—H3B	118 (6)
O23—S2—O21	111.99 (17)	C3—N3—H3C	113 (5)
O23—S2—O22	105.65 (16)	H3A—N3—H3C	111 (6)
O21—S2—O22	110.84 (18)	H3B—N3—H3C	99 (6)
O23—S2—O24	111.67 (17)	N3—C3—C2	111.2 (4)
O21—S2—O24	106.03 (16)	N3—C3—H3D	109 (4)
O22—S2—O24	110.75 (17)	C2—C3—H3D	110 (4)
O32—S3—O31	111.4 (2)	N3—C3—H3E	109 (4)
O32—S3—O34	110.9 (2)	C2—C3—H3E	118 (4)
O31—S3—O34	109.50 (18)	H3D—C3—H3E	99 (5)
O32—S3—O33	110.8 (2)		

Symmetry codes: (i) $x+1, y, z$; (ii) $x, -y+1/2, z+1/2$; (iii) $x, -y+1/2, z-1/2$; (iv) $x-1, y, z$; (v) $-x, -y, -z+1$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H11···O32	0.72 (8)	2.53 (8)	2.974 (6)	121 (7)
O1—H12···O13	0.78 (6)	1.92 (6)	2.674 (5)	162 (6)
O2—H21···O3	1.00 (12)	1.89 (12)	2.858 (7)	163 (9)
O2—H22···O21^{ii}	0.77 (8)	2.29 (8)	2.905 (6)	137 (7)
O3—H31···O11^{vi}	0.87 (7)	1.95 (8)	2.795 (5)	165 (7)

O3—H32···O12^{vii}	0.80 (8)	2.00 (8)	2.766 (5)	162 (8)
N1—H1*A*···O33	0.87 (8)	2.48 (8)	3.291 (5)	155 (6)
N1—H1*B*···O3	0.88 (7)	1.92 (7)	2.758 (6)	158 (6)
N1—H1*C*···O13^{v}	0.99 (9)	1.85 (9)	2.841 (6)	176 (7)
N2—H2*A*···O24	0.76 (7)	2.21 (7)	2.976 (5)	177 (7)
N2—H2*B*···O22^{viii}	0.83 (8)	2.17 (8)	2.967 (6)	162 (7)
N2—H2*C*···O34^{ix}	0.94 (7)	2.20 (6)	3.020 (5)	146 (5)
N3—H3*A*···O2^{x}	0.85 (7)	2.12 (7)	2.901 (8)	153 (6)
N3—H3*B*···O11	0.90 (7)	1.95 (8)	2.847 (6)	175 (6)
N3—H3*C*···O33	0.87 (7)	2.20 (7)	3.066 (5)	173 (6)

Symmetry codes: (ii) x, $-y+1/2$, $z+1/2$; (v) $-x$, $-y$, $-z+1$; (vi) x, y, $z+1$; (vii) $-x+1$, $-y$, $-z+1$; (viii) $x-1$, $-y+1/2$, $z-1/2$; (ix) x, y, $z-1$; (x) $x-1$, y, $z-1$.