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Poly[diaquabis(μ_3-1*H*-imidazole-4,5-dicarboxylato)(μ_2-sulfato)-diytterbium(III)]

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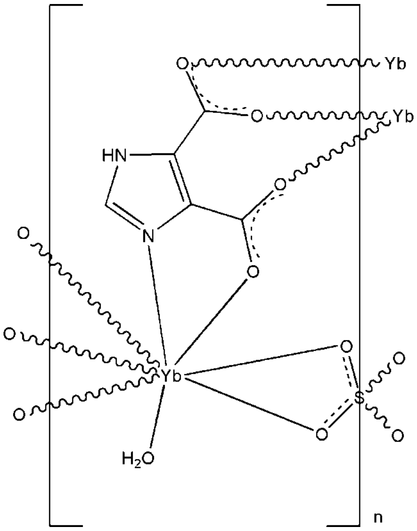
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.007 Å; R factor = 0.020; wR factor = 0.047; data-to-parameter ratio = 10.2.

In the title compound, $[Yb_2(C_5H_2N_2O_4)_2(SO_4)(H_2O)_2]_n$, the Yb^{III} ion is eight-coordinated by four O atoms and one N atom from three imidazole-4,5-dicarboxylate ligands, two O atoms from one SO_4^{2-} anion (site symmetry 2), as well as one O atom of a water molecule, giving a bicapped trigonal–prismatic coordination geometry. The metal coordination units are connected by bridging imidazole-4,5-dicarboxylate and sulfate ligands, generating a heterometallic layer. The layers are stacked along the a axis *via* N—H$\cdots$O, O—H$\cdots$O, and C—H$\cdots$O hydrogen-bonding interactions, generating a three-dimensional framework.

Related literature

For the application of multifunctional organic ligands containing O- and N-donors in the design of metal-organic frameworks, see: Cheng *et al.* (2006); Kuang *et al.* (2007); Sun *et al.* (2006); Zhu *et al.* (2010).



Experimental

Crystal data

$[Yb_2(C_5H_2N_2O_4)_2(SO_4)(H_2O)_2]$
M_r = 786.35
Monoclinic, $C2/c$
a = 21.1089 (14) Å
b = 6.5584 (4) Å
c = 12.8766 (9) Å
β = 105.874 (1)°
V = 1714.7 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 11.05 mm^{-1}
T = 296 K
0.20 × 0.18 × 0.15 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) T_{min} = 0.126, T_{max} = 0.191
4239 measured reflections
1534 independent reflections
1392 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.047
S = 1.09
1534 reflections
150 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.99 e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H1$\cdots$O6^{i}	0.87 (5)	2.09 (3)	2.925 (6)	161 (5)
O1W—H2$W\cdots$O2^{ii}	0.82 (2)	1.94 (3)	2.693 (5)	151 (5)
O1W—H1$W\cdots$O3^{iii}	0.82 (6)	2.24 (4)	2.896 (5)	138 (5)
O1W—H1$W\cdots$O4^{iii}	0.82 (6)	2.51 (6)	3.308 (5)	167 (5)
C5—H5$\cdots$O5^{iv}	0.93	2.52	3.347 (6)	149

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); 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: *SHELXL97*.

The authors acknowledge South China Normal University for supporting this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2472).

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supporting information

Acta Cryst. (2011). E**67**, m1683 [https://doi.org/10.1107/S1600536811045673]

Poly[diaquabis(μ_3-1*H*-imidazole-4,5-dicarboxylato)(μ_2-sulfato)diytterbium(III)]

Li-Cai Zhu

S1. Comment

In the past few years, the application of multifunctional organic ligands containing O– and N–donors to design metal-organic frameworks are of increasing interest, not only because of their impressive topological structures, but also due to their versatile applications in ion exchange, magnetism, bimetallic catalysis and luminescent probe (Cheng *et al.*, 2006; Kuang *et al.*, 2007; Sun *et al.*, 2006; Zhu *et al.*, 2010). As an extension of this research, the structure of the title compound, a new metal-organic framework, has been determined which is presented in this artcle.

The asymmetric unite of the title compound (Fig. 1), contains a Yb^{III} ion, an imidazole-4,5-dicarboxylate ligand, a half SO_4^{2-} anion, and a coordinated water molecule. The Yb^{III} ion is eight-coordinated by four O atoms and a N atom from three imidazole-4,5-dicarboxylate ligands, two O atoms from a SO_4^{2-} anion as well as a coordinated water molecule, giving a bicapped trigonal prismatic coordination geometry. The metal coordination units are connected by bridging imidazole-4,5-dicarboxylate and sulfate ligands, generating a two-dimensional heterometallic layer. The two-dimensional layers are stacked along *a* axis *via* N—H···O, O—H···O, and C—H···O hydrogen-bonding interactions to generate the three-dimensional framework (Table 1 and Fig. 2).

S2. Experimental

A mixture of Yb_2O_3 (0.099 g, 0.25 mmol), imidazole-4,5-dicarboxylic acid (0.156 g, 1 mmol), and H_2O (7 ml) was sealed in a 20 ml Teflon-lined reaction vessel at 443 K for 5 days then slowly cooled to room temperature. The product was collected by filtration, washed with water and air-dried. Colorless block crystals suitable for X-ray analysis were obtained.

S3. Refinement

H atoms bonded to C atoms were positioned geometrically and refined as riding, with C—H = 0.93 Å and U_{iso}(H) = 1.2 U_{eq}(C). H atoms bonded to N atoms water molecules were found from difference Fourier maps and refined isotropically with a restraint of N—H = 0.87 Å, O—H = 0.82 Å and U_{iso}(H) = 1.5 U_{eq}(N, O).

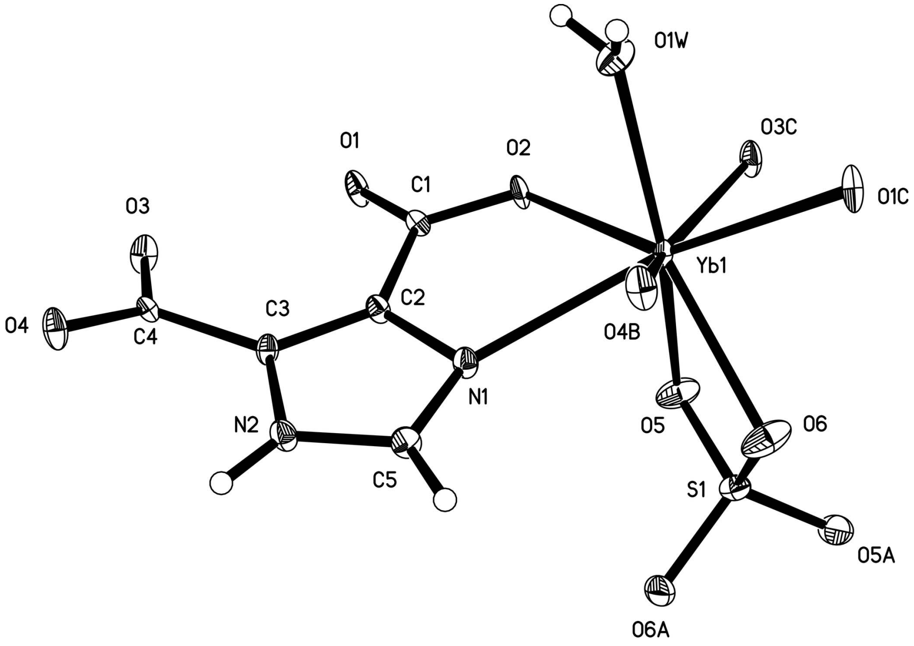

Figure 1

The molecular structure of the title comples showing atomic-numbering scheme and displacement ellipsoids drawn at 30% probability level. Symmetry codes: A = 1 - *x*, *y*, 1.5 - *z*; B = *x*, 1 - *y*, -1/2 + *z*; C = *x*, -*y*, -1/2 + *z*.

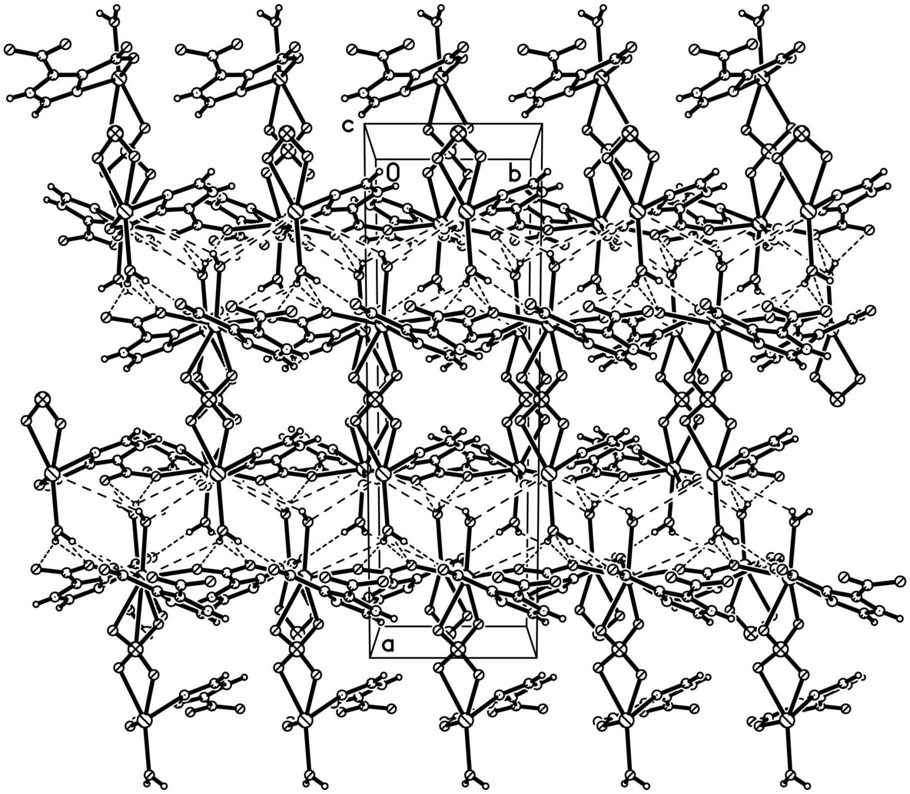

Figure 2

A view of the three-dimensional structure of the title compound, the hydrogen bonding interactions have been drawn as broken lines.

Poly[diaquabis(μ_3-1*H*-imidazole-4,5-dicarboxylato)(μ_2- sulfato)diytterbium(III)]

Crystal data

$[Yb_2(C_5H_2N_2O_4)_2(SO_4)(H_2O)_2]$
M_r = 786.35
Monoclinic, $C2/c$
Hall symbol: -C 2yc
a = 21.1089 (14) Å
b = 6.5584 (4) Å
c = 12.8766 (9) Å
β = 105.874 (1)°
V = 1714.7 (2) Å^3
Z = 4
$F(000)$ = 1456
D_x = 3.046 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2574 reflections
θ = 3.3–28.0°
μ = 11.05 mm^{-1}
T = 296 K
Block, colorless
0.20 × 0.18 × 0.15 mm

Data collection

Bruker APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scan
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min}$ = 0.126, $T_{\max}$ = 0.191
4239 measured reflections
1534 independent reflections
1392 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.023
$\theta_{\max}$ = 25.2°, $\theta_{\min}$ = 2.0°
$h = -25 \rightarrow 22$
$k = -7 \rightarrow 7$
$l = -14 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.047
S = 1.09
1534 reflections
150 parameters
4 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0216P)^2 + 5.6648P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 0.66 e Å^{-3}
$\Delta\rho_{\min}$ = −0.99 e Å^{-3}

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	$U_{\rm iso}$*/$U_{\rm eq}$
Yb1	0.355312 (10)	0.07841 (3)	0.714967 (16)	0.01086 (9)
S1	0.5000	0.0299 (3)	0.7500	0.0182 (4)
C1	0.3390 (2)	0.0272 (7)	0.9554 (4)	0.0134 (10)

C2	0.3697 (2)	0.2318 (7)	0.9654 (4)	0.0148 (10)
C3	0.3764 (2)	0.3838 (7)	1.0402 (4)	0.0145 (10)
C4	0.3492 (2)	0.4128 (7)	1.1340 (4)	0.0128 (10)
C5	0.4168 (2)	0.4805 (8)	0.9077 (4)	0.0179 (11)
H5	0.4371	0.5603	0.8664	0.022*
N1	0.3942 (2)	0.2930 (6)	0.8817 (3)	0.0165 (9)
N2	0.4065 (2)	0.5391 (6)	1.0009 (3)	0.0183 (10)
H1	0.420 (3)	0.648 (6)	1.039 (4)	0.027*
O1	0.32418 (19)	−0.0583 (5)	1.0318 (3)	0.0220 (9)
O2	0.32955 (18)	−0.0546 (5)	0.8633 (3)	0.0192 (8)
O3	0.33204 (18)	0.2591 (5)	1.1767 (3)	0.0219 (8)
O4	0.34067 (19)	0.5917 (5)	1.1620 (3)	0.0219 (8)
O5	0.45816 (18)	−0.0925 (5)	0.7997 (3)	0.0304 (10)
O6	0.45308 (19)	0.1572 (6)	0.6710 (3)	0.0349 (10)
O1W	0.24374 (18)	0.1038 (6)	0.6799 (3)	0.0250 (9)
H2W	0.224 (2)	0.201 (6)	0.646 (4)	0.037*
H1W	0.221 (3)	0.075 (8)	0.720 (4)	0.037*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Yb1	0.01712 (13)	0.00743 (13)	0.00944 (13)	0.00016 (8)	0.00604 (9)	0.00022 (8)
S1	0.0156 (9)	0.0145 (9)	0.0250 (10)	0.000	0.0063 (8)	0.000
C1	0.017 (2)	0.010 (2)	0.012 (3)	0.0000 (19)	0.001 (2)	0.001 (2)
C2	0.021 (2)	0.012 (3)	0.011 (2)	−0.003 (2)	0.0040 (19)	0.000 (2)
C3	0.021 (3)	0.013 (2)	0.011 (2)	0.002 (2)	0.006 (2)	0.001 (2)
C4	0.017 (2)	0.010 (3)	0.010 (2)	−0.0018 (19)	0.002 (2)	−0.0017 (19)
C5	0.025 (3)	0.015 (3)	0.015 (3)	−0.005 (2)	0.008 (2)	0.003 (2)
N1	0.024 (2)	0.012 (2)	0.016 (2)	−0.0023 (17)	0.0082 (18)	−0.0010 (17)
N2	0.027 (2)	0.014 (2)	0.016 (2)	−0.0064 (19)	0.0084 (19)	−0.0018 (18)
O1	0.042 (2)	0.0151 (19)	0.0107 (19)	−0.0090 (16)	0.0095 (17)	−0.0001 (15)
O2	0.033 (2)	0.0155 (19)	0.0110 (18)	−0.0084 (15)	0.0090 (15)	−0.0058 (15)
O3	0.037 (2)	0.0144 (19)	0.0182 (18)	0.0042 (16)	0.0134 (16)	0.0061 (16)
O4	0.039 (2)	0.0103 (19)	0.018 (2)	0.0028 (16)	0.0105 (17)	−0.0009 (14)
O5	0.021 (2)	0.030 (2)	0.041 (3)	0.0019 (16)	0.0104 (18)	0.0155 (19)
O6	0.023 (2)	0.044 (2)	0.041 (3)	0.0071 (19)	0.0135 (19)	0.027 (2)
O1W	0.024 (2)	0.023 (2)	0.031 (2)	0.0066 (17)	0.0129 (18)	0.0083 (17)

Geometric parameters (Å, º)

Yb1—O4$^{\text{i}}$	2.264 (3)	C1—C2	1.481 (6)
Yb1—O1$^{\text{ii}}$	2.272 (3)	C2—C3	1.367 (7)
Yb1—O1W	2.280 (4)	C2—N1	1.377 (6)
Yb1—O3$^{\text{ii}}$	2.291 (3)	C3—N2	1.369 (6)
Yb1—O2	2.297 (3)	C3—C4	1.485 (7)
Yb1—O6	2.342 (4)	C4—O3	1.249 (6)
Yb1—O5	2.421 (4)	C4—O4	1.255 (5)
Yb1—N1	2.510 (4)	C5—N1	1.328 (6)

Yb1—S1	2.9798 (3)	C5—N2	1.334 (7)
S1—O5^{iii}	1.464 (4)	C5—H5	0.9300
S1—O5	1.464 (4)	N2—H1	0.87 (5)
S1—O6^{iii}	1.470 (4)	O1—Yb1^{iv}	2.272 (3)
S1—O6	1.470 (4)	O3—Yb1^{iv}	2.291 (3)
S1—Yb1^{iii}	2.9798 (3)	O4—Yb1^{v}	2.264 (3)
C1—O1	1.244 (6)	O1W—H2W	0.82 (2)
C1—O2	1.266 (6)	O1W—H1W	0.82 (6)

O4^{i}—Yb1—O1^{ii}	76.46 (12)	O6^{iii}—S1—O6	110.8 (4)
O4^{i}—Yb1—O1W	79.74 (14)	O5^{iii}—S1—Yb1	135.09 (15)
O1^{ii}—Yb1—O1W	78.99 (15)	O5—S1—Yb1	53.75 (14)
O4^{i}—Yb1—O3^{ii}	148.74 (13)	O6^{iii}—S1—Yb1	120.86 (16)
O1^{ii}—Yb1—O3^{ii}	74.75 (12)	O6—S1—Yb1	50.65 (15)
O1W—Yb1—O3^{ii}	83.05 (14)	O5^{iii}—S1—Yb1^{iii}	53.75 (14)
O4^{i}—Yb1—O2	124.73 (12)	O5—S1—Yb1^{iii}	135.09 (15)
O1^{ii}—Yb1—O2	140.73 (13)	O6^{iii}—S1—Yb1^{iii}	50.65 (15)
O1W—Yb1—O2	74.07 (14)	O6—S1—Yb1^{iii}	120.86 (16)
O3^{ii}—Yb1—O2	74.10 (12)	Yb1—S1—Yb1^{iii}	167.75 (7)
O4^{i}—Yb1—O6	76.89 (14)	O1—C1—O2	122.7 (4)
O1^{ii}—Yb1—O6	77.64 (14)	O1—C1—C2	122.6 (4)
O1W—Yb1—O6	150.11 (14)	O2—C1—C2	114.7 (4)
O3^{ii}—Yb1—O6	108.21 (14)	C3—C2—N1	110.6 (4)
O2—Yb1—O6	135.20 (13)	C3—C2—C1	132.9 (5)
O4^{i}—Yb1—O5	127.57 (13)	N1—C2—C1	116.5 (4)
O1^{ii}—Yb1—O5	114.21 (14)	C2—C3—N2	104.5 (4)
O1W—Yb1—O5	150.85 (13)	C2—C3—C4	132.8 (4)
O3^{ii}—Yb1—O5	76.28 (13)	N2—C3—C4	121.8 (4)
O2—Yb1—O5	80.58 (13)	O3—C4—O4	123.2 (5)
O6—Yb1—O5	58.02 (13)	O3—C4—C3	118.5 (4)
O4^{i}—Yb1—N1	72.99 (13)	O4—C4—C3	118.1 (4)
O1^{ii}—Yb1—N1	148.68 (12)	N1—C5—N2	111.0 (4)
O1W—Yb1—N1	101.95 (14)	N1—C5—H5	124.5
O3^{ii}—Yb1—N1	136.57 (13)	N2—C5—H5	124.5
O2—Yb1—N1	66.31 (12)	C5—N1—C2	105.0 (4)
O6—Yb1—N1	88.79 (15)	C5—N1—Yb1	138.2 (3)
O5—Yb1—N1	80.25 (14)	C2—N1—Yb1	113.6 (3)
O4^{i}—Yb1—S1	101.39 (10)	C5—N2—C3	109.0 (4)
O1^{ii}—Yb1—S1	98.30 (10)	C5—N2—H1	130 (4)
O1W—Yb1—S1	176.78 (11)	C3—N2—H1	121 (4)
O3^{ii}—Yb1—S1	94.59 (10)	C1—O1—Yb1^{iv}	141.5 (3)
O2—Yb1—S1	107.44 (9)	C1—O2—Yb1	127.3 (3)
O6—Yb1—S1	29.03 (9)	C4—O3—Yb1^{iv}	143.5 (3)
O5—Yb1—S1	29.18 (9)	C4—O4—Yb1^{v}	164.1 (3)
N1—Yb1—S1	81.27 (10)	S1—O5—Yb1	97.06 (18)
O5^{iii}—S1—O5	113.5 (3)	S1—O6—Yb1	100.32 (19)
O5^{iii}—S1—O6^{iii}	103.9 (2)	Yb1—O1W—H2W	121 (4)
O5—S1—O6^{iii}	112.4 (2)	Yb1—O1W—H1W	128 (4)

O5^{iii}—S1—O6	112.4 (2)	H2W—O1W—H1W	102 (3)
O5—S1—O6	103.9 (2)		

Symmetry codes: (i) $x, -y+1, z-1/2$; (ii) $x, -y, z-1/2$; (iii) $-x+1, y, -z+3/2$; (iv) $x, -y, z+1/2$; (v) $x, -y+1, z+1/2$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1···O6^{v}	0.87 (5)	2.09 (3)	2.925 (6)	161 (5)
O1*W*—H2*W*···O2^{vi}	0.82 (2)	1.94 (3)	2.693 (5)	151 (5)
O1*W*—H1*W*···O3^{vii}	0.82 (6)	2.24 (4)	2.896 (5)	138 (5)
O1*W*—H1*W*···O4^{vii}	0.82 (6)	2.51 (6)	3.308 (5)	167 (5)
C5—H5···O5^{viii}	0.93	2.52	3.347 (6)	149

Symmetry codes: (v) $x, -y+1, z+1/2$; (vi) $-x+1/2, y+1/2, -z+3/2$; (vii) $-x+1/2, -y+1/2, -z+2$; (viii) $x, y+1, z$.