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## Tris(ethylenediamine)cobalt(II) sulfate

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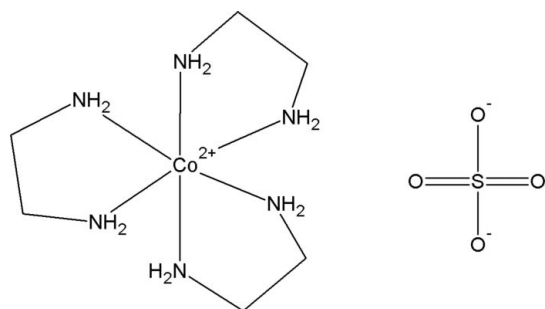
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Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å; disorder in main residue;  $R$  factor = 0.028;  $wR$  factor = 0.069; data-to-parameter ratio = 14.6.

The structure of the title compound,  $[\text{Co}^{\text{II}}(\text{C}_2\text{H}_8\text{N}_2)_3]\text{SO}_4$ , the cobalt example of  $[M(\text{C}_2\text{H}_8\text{N}_2)_3]\text{SO}_4$ , is reported. The Co and S atoms are located at the  $2d$  and  $2c$  Wyckoff sites (point symmetry 32), respectively. The Co atom is coordinated by six N atoms of three chelating ethylenediamine molecules generated from half of the ethylenediamine molecule in the asymmetric unit. The O atoms of the sulfate anion are disordered mostly over two crystallographic sites. The third disorder site of O (site symmetry 3) has a site occupancy approaching zero. The H atoms of the ethylenediamine molecules interact with the sulfate anions *via* intermolecular N—H $\cdots$ O hydrogen-bonding interactions.

## Related literature

For isostructural  $[M(\text{C}_2\text{H}_8\text{N}_2)_3]\text{SO}_4$  complexes, see: Haque *et al.* (1970); Cullen & Lingafelter (1970); Daniels *et al.* (1995); Lu (2009) for the nickel, copper, vanadium and manganese analogues, respectively.



## Experimental

## Crystal data

$[\text{Co}(\text{C}_2\text{H}_8\text{N}_2)_3]\text{SO}_4$   
 $M_r = 335.30$   
 Trigonal,  $P\bar{3}1c$

$a = 8.9920$  (2) Å  
 $c = 9.5927$  (3) Å  
 $V = 671.71$  (3) Å<sup>3</sup>

$Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 1.45$  mm<sup>-1</sup>

$T = 298$  K  
 $0.48 \times 0.22 \times 0.20$  mm

## Data collection

Bruker SMART CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\text{min}} = 0.543$ ,  $T_{\text{max}} = 0.760$

3638 measured reflections  
 688 independent reflections  
 589 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.027$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.028$   
 $wR(F^2) = 0.069$   
 $S = 1.06$   
 688 reflections  
 47 parameters

16 restraints  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.25$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.29$  e Å<sup>-3</sup>

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A $\cdots$ O3 <sup>i</sup>	0.90	2.13	2.889 (12)	142
N1—H1A $\cdots$ O1 <sup>i</sup>	0.90	2.15	3.049 (7)	176
N1—H1A $\cdots$ O2 <sup>ii</sup>	0.90	2.22	3.054 (8)	155
N1—H1A $\cdots$ O2 <sup>iii</sup>	0.90	2.32	3.104 (11)	145
N1—H1B $\cdots$ O2 <sup>iv</sup>	0.90	1.98	2.843 (6)	161
N1—H1B $\cdots$ O1	0.90	2.48	3.353 (14)	165
N1—H1B $\cdots$ O1 <sup>v</sup>	0.90	2.52	3.256 (10)	139

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

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008) and *WinGX* (Farrugia, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008) and *WinGX* (Farrugia, 1999); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2667).

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## supporting information

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**Tris(ethylenediamine)cobalt(II) sulfate****Bunlawee Yotnoi, Athittaya Seeharaj, Yothin Chimupala and Apinpus Rujiwatra****S1. Comment**

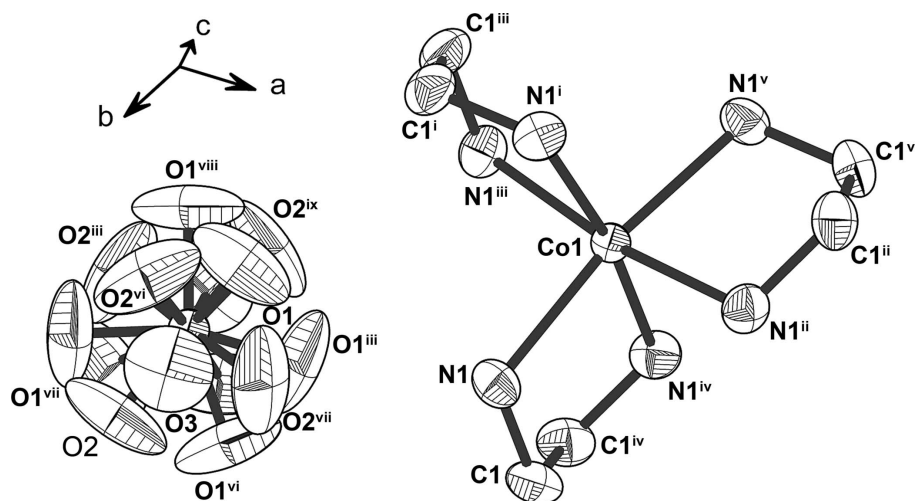
The title complex,  $[\text{Co}^{\text{II}}(\text{C}_2\text{H}_8\text{N}_2)_3]\text{SO}_4$  (Fig. 1), is isostructural to the earlier reported  $[\text{Ni}^{\text{II}}(\text{C}_2\text{H}_8\text{N}_2)_3]\text{SO}_4$  (Haque *et al.*, 1970),  $[\text{V}^{\text{II}}(\text{C}_2\text{H}_8\text{N}_2)_3]\text{SO}_4$  (Daniels *et al.*, 1995),  $[\text{Mn}^{\text{II}}(\text{C}_2\text{H}_8\text{N}_2)_3]\text{SO}_4$  (Lu, 2009) and  $[\text{Cu}^{\text{II}}(\text{C}_2\text{H}_8\text{N}_2)_3]\text{SO}_4$  (Cullen & Lingafelter, 1970) complexes, constituting the  $[\text{M}^{\text{II}}(\text{C}_2\text{H}_8\text{N}_2)_3]\text{SO}_4$  series. The  $[\text{M}^{\text{II}}(\text{C}_2\text{H}_8\text{N}_2)_3]\text{SO}_4$  structures crystallize in the same trigonal space group of  $\bar{P}31c$  with quite similar cell parameters. Likewise, the metal and sulfur atoms are positioned in the same crystallographic sites;  $\text{M}^{\text{II}}$  on the  $2d$  and S on the  $2c$  Wyckoff sites (each with point symmetry  $32$ ). The disorder about the six-fold rotation axis found in the sulfate anion is intriguingly common in each structure, although the number of unique O atoms varies from two to four. In the structure of  $[\text{Co}^{\text{II}}(\text{C}_2\text{H}_8\text{N}_2)_3]\text{SO}_4$ , the O atoms were refined as being disordered over three crystallographic sites, although the site occupancy of O3 located on the  $4f$  Wyckoff site approaches zero. The bond length associated with this O3 atom (S1—O3; 1.382 (16) Å) is notably shorter than the other S—O bonds (1.431 (5)—1.445 (5) Å). The disordered sulfate anions are linked to the  $[\text{Co}^{\text{II}}(\text{C}_2\text{H}_8\text{N}_2)_3]^{2+}$  cations by hydrogen bonding interactions of N—H $\cdots$ O type to form a hydrogen-bonding supramolecular network. The hydrogen bonding geometries are consistent with those of the previously reported  $[\text{M}^{\text{II}}(\text{C}_2\text{H}_8\text{N}_2)_3]\text{SO}_4$  complexes.

**S2. Experimental**

Orange blocks of the title complex were synthesized and grown from the solvothermal reaction of  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  (1.34 mmol),  $\text{NH}_2\text{SO}_3\text{H}$  (1.34 mmol),  $\text{NH}_2\text{C}_2\text{H}_4\text{NH}_2$  (3.89 mmol) in ethylene glycol (160 mmol), conducted at 453 K for 72 h.

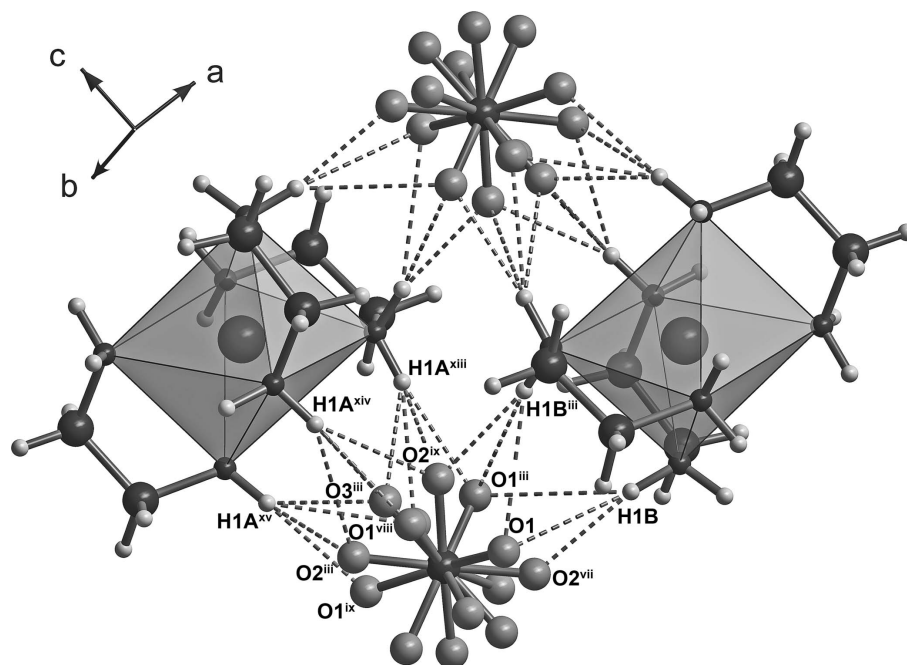
**S3. Refinement**

The O atoms were positioned from a difference Fourier map, and refined with restraints using commands SUMP, SADI and SIMU in *SHELXL* (Sheldrick, 2008). Although there was an indication for further splitting of the O2 atom, after the final cycles of refinement, such action did not give a better result. All H-atoms were treated as riding groups on the bonded atoms, with C—H = 0.97 Å and N—H 0.90 Å, and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{equiv}}(\text{C}, \text{N})$ .



**Figure 1**

View of the title complex showing atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level. [Symmetry codes: (i)  $-y+1, x-y, z$ ; (ii)  $-x+y+1, -x+1, z$ ; (iii)  $-y+1, -x+1, -z+1/2$ ; (iv)  $-x+y+1, y, -z+1/2$ ; (v)  $x, x-y, -z+1/2$ ; (vi)  $-y+1, x-y+1, z$ ; (vii)  $-x+y, -x+1, z$ ; (viii)  $-x+y, y, -z+1/2$ ; (ix)  $x, x-y+1, -z+1/2$ ]. Hydrogen atoms are omitted.



**Figure 2**

View of the hydrogen bonding interactions (dotted lines) between the disordered sulfate O atoms and the amino-H atoms of the  $[\text{Co}^{\text{II}}(\text{C}_2\text{H}_8\text{N}_2)]^{2+}$  cations. [Symmetry codes: (ii)  $-x+y+1, -x+1, z$ ; (iii)  $-y+1, -x+1, -z+1/2$ ; (viii)  $-x+y, y, -z+1/2$ ; (ix)  $x, x-y+1, -z+1/2$ ; (xiii)  $y, x, z+1/2$ ; (xiv)  $-y+x, -y+1, -z+1/2$ ; (xv)  $-x+1, -x+y+1, z+1/2$ ].

Tris(ethylenediamine)cobalt(II) sulfate

Crystal data

[Co(C<sub>2</sub>H<sub>8</sub>N<sub>2</sub>)<sub>3</sub>]<sub>2</sub>SO<sub>4</sub>  
*M<sub>r</sub>* = 335.30  
 Trigonal,  $P\bar{3}1c$   
 Hall symbol: -P 3 2c  
*a* = 8.9920 (2) Å  
*c* = 9.5927 (3) Å  
*V* = 671.71 (3) Å<sup>3</sup>  
*Z* = 2  
*F*(000) = 354

*D<sub>x</sub>* = 1.658 Mg m<sup>-3</sup>  
 Mo *Kα* radiation,  $\lambda$  = 0.71073 Å  
 Cell parameters from 589 reflections  
 $\theta$  = 2.6–31.0°  
 $\mu$  = 1.45 mm<sup>-1</sup>  
*T* = 298 K  
 Block, orange  
 0.48 × 0.22 × 0.20 mm

Data collection

Bruker SMART CCD area-detector  
 diffractometer  
 Radiation source: fine-focus sealed tube  
 Graphite monochromator  
 ? scan  
 Absorption correction: multi-scan  
 (SADABS; Sheldrick, 1996)  
*T<sub>min</sub>* = 0.543, *T<sub>max</sub>* = 0.760

3638 measured reflections  
 688 independent reflections  
 589 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.027  
 $\theta_{\max}$  = 31.0°,  $\theta_{\min}$  = 2.6°  
*h* = -8→10  
*k* = -11→11  
*l* = -11→13

Refinement

Refinement on *F*<sup>2</sup>  
 Least-squares matrix: full  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.028  
*wR*(*F*<sup>2</sup>) = 0.069  
*S* = 1.06  
 688 reflections  
 47 parameters  
 16 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/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0354*P*)<sup>2</sup> + 0.1217*P*]  
 where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3  
 (Δ/σ)<sub>max</sub> < 0.001  
 Δρ<sub>max</sub> = 0.25 e Å<sup>-3</sup>  
 Δρ<sub>min</sub> = -0.29 e Å<sup>-3</sup>

Special details

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

**Refinement.** Refinement of *F*<sup>2</sup> against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on *F*<sup>2</sup>, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative *F*<sup>2</sup>. The threshold expression of *F*<sup>2</sup> > σ(*F*<sup>2</sup>) 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*<sup>2</sup> 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<sup>2</sup>)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>iso</sub></i> */ <i>U<sub>eq</sub></i>	Occ. (<1)
Co1	0.6667	0.3333	0.2500	0.02175 (16)	
N1	0.68784 (18)	0.54599 (18)	0.12760 (13)	0.0332 (3)	
H1A	0.6936	0.5265	0.0363	0.040*	
H1B	0.5954	0.5579	0.1418	0.040*	
S1	0.3333	0.6667	0.2500	0.0240 (2)	

C1	0.8446 (2)	0.7024 (2)	0.17145 (19)	0.0388 (4)	
H1C	0.8405	0.8031	0.1409	0.047*	
H1D	0.9444	0.7056	0.1297	0.047*	
O1	0.3029 (19)	0.5088 (9)	0.1852 (8)	0.096 (3)	0.319 (8)
O2	0.339 (2)	0.7851 (9)	0.1475 (6)	0.096 (4)	0.316 (9)
O3	0.3333	0.6667	0.1059 (16)	0.086 (8)	0.094 (10)

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Co1	0.0226 (2)	0.0226 (2)	0.0201 (2)	0.01129 (10)	0.000	0.000
N1	0.0410 (8)	0.0330 (7)	0.0283 (7)	0.0204 (6)	-0.0033 (5)	0.0031 (5)
S1	0.0243 (3)	0.0243 (3)	0.0233 (4)	0.01215 (14)	0.000	0.000
C1	0.0445 (10)	0.0267 (8)	0.0413 (9)	0.0147 (7)	0.0058 (7)	0.0076 (6)
O1	0.185 (9)	0.051 (4)	0.063 (4)	0.069 (5)	-0.012 (5)	-0.016 (3)
O2	0.194 (12)	0.055 (4)	0.044 (3)	0.067 (5)	-0.012 (4)	0.012 (3)
O3	0.118 (11)	0.118 (11)	0.021 (11)	0.059 (5)	0.000	0.000

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Co1—N1 <sup>i</sup>	2.1696 (13)	S1—O2 <sup>vi</sup>	1.431 (5)
Co1—N1 <sup>ii</sup>	2.1696 (13)	S1—O2 <sup>v</sup>	1.431 (5)
Co1—N1 <sup>iii</sup>	2.1696 (13)	S1—O2 <sup>vii</sup>	1.431 (5)
Co1—N1 <sup>iv</sup>	2.1696 (13)	S1—O2 <sup>viii</sup>	1.431 (5)
Co1—N1	2.1696 (13)	S1—O2 <sup>ix</sup>	1.431 (5)
Co1—N1 <sup>v</sup>	2.1696 (13)	S1—O1 <sup>ix</sup>	1.445 (5)
N1—C1	1.469 (2)	S1—O1 <sup>viii</sup>	1.445 (5)
N1—H1A	0.9000	S1—O1 <sup>vi</sup>	1.445 (5)
N1—H1B	0.9000	S1—O1 <sup>vii</sup>	1.445 (5)
S1—O3	1.382 (16)	C1—C1 <sup>iv</sup>	1.512 (4)
S1—O3 <sup>v</sup>	1.382 (16)	C1—H1C	0.9700
S1—O2	1.431 (5)	C1—H1D	0.9700
N1 <sup>i</sup> —Co1—N1 <sup>ii</sup>	80.49 (7)	O2 <sup>viii</sup> —S1—O1 <sup>viii</sup>	110.7 (4)
N1 <sup>i</sup> —Co1—N1 <sup>iii</sup>	93.48 (5)	O2 <sup>ix</sup> —S1—O1 <sup>viii</sup>	138.0 (11)
N1 <sup>ii</sup> —Co1—N1 <sup>iii</sup>	93.17 (8)	O1 <sup>ix</sup> —S1—O1 <sup>viii</sup>	63.4 (8)
N1 <sup>i</sup> —Co1—N1 <sup>iv</sup>	93.17 (8)	O3—S1—O1 <sup>vi</sup>	64.5 (3)
N1 <sup>ii</sup> —Co1—N1 <sup>iv</sup>	93.48 (5)	O3 <sup>v</sup> —S1—O1 <sup>vi</sup>	115.5 (3)
N1 <sup>iii</sup> —Co1—N1 <sup>iv</sup>	171.28 (7)	O2—S1—O1 <sup>vi</sup>	57.2 (5)
N1 <sup>i</sup> —Co1—N1	93.48 (5)	O2 <sup>vi</sup> —S1—O1 <sup>vi</sup>	110.7 (4)
N1 <sup>ii</sup> —Co1—N1	171.28 (8)	O2 <sup>v</sup> —S1—O1 <sup>vi</sup>	138.0 (11)
N1 <sup>iii</sup> —Co1—N1	93.48 (5)	O2 <sup>vii</sup> —S1—O1 <sup>vi</sup>	69.9 (6)
N1 <sup>iv</sup> —Co1—N1	80.49 (7)	O2 <sup>viii</sup> —S1—O1 <sup>vi</sup>	45.7 (4)
N1 <sup>i</sup> —Co1—N1 <sup>v</sup>	171.28 (8)	O2 <sup>ix</sup> —S1—O1 <sup>vi</sup>	119.2 (10)
N1 <sup>ii</sup> —Co1—N1 <sup>v</sup>	93.48 (5)	O1 <sup>ix</sup> —S1—O1 <sup>vi</sup>	93.3 (11)
N1 <sup>iii</sup> —Co1—N1 <sup>v</sup>	80.49 (7)	O1 <sup>viii</sup> —S1—O1 <sup>vi</sup>	102.9 (4)
N1 <sup>iv</sup> —Co1—N1 <sup>v</sup>	93.48 (5)	O3—S1—O1 <sup>vii</sup>	115.5 (3)
N1—Co1—N1 <sup>v</sup>	93.17 (8)	O3 <sup>v</sup> —S1—O1 <sup>vii</sup>	64.5 (3)

C1—N1—Co1	107.94 (10)	O2—S1—O1 <sup>vii</sup>	138.0 (11)
C1—N1—H1A	110.1	O2 <sup>vi</sup> —S1—O1 <sup>vii</sup>	69.9 (6)
Co1—N1—H1A	110.1	O2 <sup>v</sup> —S1—O1 <sup>vii</sup>	57.2 (5)
C1—N1—H1B	110.1	O2 <sup>vii</sup> —S1—O1 <sup>vii</sup>	110.7 (4)
Co1—N1—H1B	110.1	O2 <sup>viii</sup> —S1—O1 <sup>vii</sup>	119.2 (10)
H1A—N1—H1B	108.4	O2 <sup>ix</sup> —S1—O1 <sup>vii</sup>	45.7 (4)
O3—S1—O3 <sup>v</sup>	180.000 (3)	O1 <sup>ix</sup> —S1—O1 <sup>vii</sup>	102.9 (4)
O3—S1—O2	46.6 (3)	O1 <sup>viii</sup> —S1—O1 <sup>vii</sup>	93.3 (11)
O3 <sup>v</sup> —S1—O2	133.4 (3)	O1 <sup>vi</sup> —S1—O1 <sup>vii</sup>	161.1 (12)
O3—S1—O2 <sup>vi</sup>	46.6 (3)	N1—C1—C1 <sup>iv</sup>	108.84 (12)
O3 <sup>v</sup> —S1—O2 <sup>vi</sup>	133.4 (3)	N1—C1—H1C	109.9
O2—S1—O2 <sup>vi</sup>	78.0 (5)	C1 <sup>iv</sup> —C1—H1C	109.9
O3—S1—O2 <sup>v</sup>	133.4 (3)	N1—C1—H1D	109.9
O3 <sup>v</sup> —S1—O2 <sup>v</sup>	46.6 (3)	C1 <sup>iv</sup> —C1—H1D	109.9
O2—S1—O2 <sup>v</sup>	104.4 (11)	H1C—C1—H1D	108.3
O2 <sup>vi</sup> —S1—O2 <sup>v</sup>	99.7 (7)	O2 <sup>vi</sup> —O1—O2 <sup>viii</sup>	91.9 (8)
O3—S1—O2 <sup>vii</sup>	133.4 (3)	O2 <sup>vi</sup> —O1—S1	66.5 (5)
O3 <sup>v</sup> —S1—O2 <sup>vii</sup>	46.6 (3)	O2 <sup>viii</sup> —O1—S1	60.9 (3)
O2—S1—O2 <sup>vii</sup>	99.7 (7)	O2 <sup>vi</sup> —O1—O1 <sup>vii</sup>	75.7 (11)
O2 <sup>vi</sup> —S1—O2 <sup>vii</sup>	176.3 (13)	O2 <sup>viii</sup> —O1—O1 <sup>vii</sup>	117.8 (4)
O2 <sup>v</sup> —S1—O2 <sup>vii</sup>	78.0 (5)	S1—O1—O1 <sup>vii</sup>	58.3 (4)
O3—S1—O2 <sup>viii</sup>	46.6 (3)	O2 <sup>vi</sup> —O1—O2 <sup>ix</sup>	108.3 (7)
O3 <sup>v</sup> —S1—O2 <sup>viii</sup>	133.4 (3)	O2 <sup>viii</sup> —O1—O2 <sup>ix</sup>	92.2 (8)
O2—S1—O2 <sup>viii</sup>	78.0 (5)	S1—O1—O2 <sup>ix</sup>	54.6 (4)
O2 <sup>vi</sup> —S1—O2 <sup>viii</sup>	78.0 (5)	O1 <sup>viii</sup> —O2—O1 <sup>vi</sup>	129.8 (7)
O2 <sup>v</sup> —S1—O2 <sup>viii</sup>	176.3 (13)	O1 <sup>viii</sup> —O2—S1	67.8 (4)
O2 <sup>vii</sup> —S1—O2 <sup>viii</sup>	104.4 (11)	O1 <sup>vi</sup> —O2—S1	61.9 (4)
O3—S1—O2 <sup>ix</sup>	133.4 (3)	O1 <sup>viii</sup> —O2—O1 <sup>ix</sup>	63.3 (12)
O3 <sup>v</sup> —S1—O2 <sup>ix</sup>	46.6 (3)	O1 <sup>vi</sup> —O2—O1 <sup>ix</sup>	87.5 (9)
O2—S1—O2 <sup>ix</sup>	176.3 (13)	S1—O2—O1 <sup>ix</sup>	55.5 (3)
O2 <sup>vi</sup> —S1—O2 <sup>ix</sup>	104.4 (11)	O1 <sup>viii</sup> —O2—O2 <sup>vi</sup>	49.8 (6)
O2 <sup>v</sup> —S1—O2 <sup>ix</sup>	78.0 (5)	O1 <sup>vi</sup> —O2—O2 <sup>vi</sup>	95.3 (5)
O2 <sup>vii</sup> —S1—O2 <sup>ix</sup>	78.0 (5)	S1—O2—O2 <sup>vi</sup>	51.0 (2)
O2 <sup>viii</sup> —S1—O2 <sup>ix</sup>	99.7 (7)	O1 <sup>ix</sup> —O2—O2 <sup>vi</sup>	91.9 (8)
O3—S1—O1 <sup>ix</sup>	115.5 (3)	O1 <sup>viii</sup> —O2—O2 <sup>viii</sup>	106.1 (5)
O3 <sup>v</sup> —S1—O1 <sup>ix</sup>	64.5 (3)	S1—O2—O2 <sup>viii</sup>	51.0 (2)
O2—S1—O1 <sup>ix</sup>	69.9 (6)	O1 <sup>ix</sup> —O2—O2 <sup>viii</sup>	102.2 (4)
O2 <sup>vi</sup> —S1—O1 <sup>ix</sup>	119.2 (10)	O2 <sup>vi</sup> —O2—O2 <sup>viii</sup>	60.000 (1)
O2 <sup>v</sup> —S1—O1 <sup>ix</sup>	45.7 (4)	O2 <sup>vi</sup> —O3—O2 <sup>viii</sup>	107.9 (8)
O2 <sup>vii</sup> —S1—O1 <sup>ix</sup>	57.2 (5)	O2 <sup>vi</sup> —O3—S1	69.0 (8)
O2 <sup>viii</sup> —S1—O1 <sup>ix</sup>	138.0 (11)	O2 <sup>viii</sup> —O3—S1	69.0 (8)
O2 <sup>ix</sup> —S1—O1 <sup>ix</sup>	110.7 (4)	O2 <sup>vi</sup> —O3—O1 <sup>viii</sup>	61.1 (6)
O3—S1—O1 <sup>viii</sup>	64.5 (3)	O2 <sup>viii</sup> —O3—O1 <sup>viii</sup>	128.2 (13)
O3 <sup>v</sup> —S1—O1 <sup>viii</sup>	115.5 (3)	S1—O3—O1 <sup>viii</sup>	59.8 (5)
O2—S1—O1 <sup>viii</sup>	45.7 (4)	O2 <sup>vi</sup> —O3—O1 <sup>vi</sup>	128.2 (13)
O2 <sup>vi</sup> —S1—O1 <sup>viii</sup>	57.2 (5)	O2 <sup>viii</sup> —O3—O1 <sup>vi</sup>	47.4 (6)

O2 <sup>v</sup> —S1—O1 <sup>viii</sup>	69.9 (6)	S1—O3—O1 <sup>vi</sup>	59.8 (5)
O2 <sup>vii</sup> —S1—O1 <sup>viii</sup>	119.2 (10)	O1 <sup>viii</sup> —O3—O1 <sup>vi</sup>	96.9 (7)

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

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N1—H1A $\cdots$ O3 <sup>x</sup>	0.90	2.13	2.889 (12)	142
N1—H1A $\cdots$ O1 <sup>x</sup>	0.90	2.15	3.049 (7)	176
N1—H1A $\cdots$ O2 <sup>xi</sup>	0.90	2.22	3.054 (8)	155
N1—H1A $\cdots$ O2 <sup>xii</sup>	0.90	2.32	3.104 (11)	145
N1—H1B $\cdots$ O2 <sup>viii</sup>	0.90	1.98	2.843 (6)	161
N1—H1B $\cdots$ O1	0.90	2.48	3.353 (14)	165
N1—H1B $\cdots$ O1 <sup>v</sup>	0.90	2.52	3.256 (10)	139

Symmetry codes: (v)  $-y+1, -x+1, -z+1/2$ ; (viii)  $-x+y, -x+1, z$ ; (x)  $-x+1, -y+1, -z$ ; (xi)  $y, -x+y, -z$ ; (xii)  $x-y+1, x, -z$ .