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Diglycinium sulfate

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The crystal structure of diglycinium sulfate (DGS), $2C_2H_6$-$NO_2^+ \cdot SO_4^{2-}$, consists of two layers of glycinium and sulfate groups inserted between a layer of glycinium cations linked by strong hydrogen bonds. The two glycinium cations have different conformations, viz. E for glycine A and Z for glycine B.

Comment

Many glycine salts and adducts exhibit interesting dielectric properties, the best known compound of this family being TGS (triglycine sulfate), which orders ferroelectrically below 322 K (Matthias *et al.*, 1956). In addition to their importance in the field of new materials chemistry (Siegel *et al.*, 1998; Baker *et al.*, 1992), hybrid compounds are of interest because of their electrical, magnetic and optical properties (Kagan *et al.*, 1999; Hill, 1998). The title compound, diglycinium sulfate (DGS), results from a search for new organic–inorganic hybrid materials (Benali-Cherif, Cherouana *et al.*, 2002; Benali-Cherif, Abouimrane *et al.*, 2002; Benali-Cherif, Bendheif *et al.*, 2002; Benali-Cherif, Benguedouar *et al.*, 2002.). The asymmetric unit of DGS contains two monoprotonated glycine molecules ($C_2H_7NO_2^+$) and one sulfate ion (SO_4^{2-}).

DGS
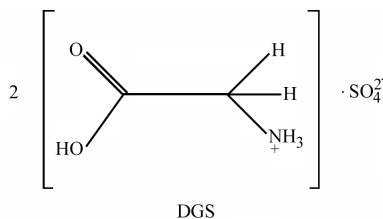
The mean bond lengths and angles in the SO_4^{2-} group are 1.472 Å and 109.5°, respectively, showing a normal tetrahedral geometry for the S atom. Interatomic distances in the glycinium cations compare well with distances observed in diglycine sulfate monohydrate (Cano & Martinez-Carrera, 1974). Although their carboxy skeletons are both planar, atom N1B is displaced from the plane by 0.170 (15) Å, whereas atom N1A is displaced by 0.075 (15) Å. The relevant torsion angles of the glycinium cations [O1A—C2A—C1A—N1A = 176.71 (15)° and N1B—C2B—C1B—O1B = 23.9 (7)°] indicate different conformations for the two glycinium cations in the asymmetric unit, viz. E for glycine A and Z for glycine B; this difference in conformation is not observed in diglycine selenate (Olejnik & Lukaszewicz, 1975). The crystal structure is composed of two layers of glycine B ions and sulfate groups inserted between layers of glycine A along their stacking direction (b axis). The layers are linked together by an intricate network of hydrogen-bond interactions. The strongest of
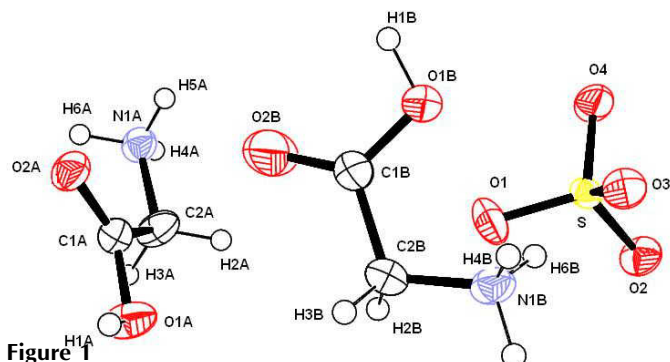
Figure 1
ORTEP-3 (Farrugia, 1997) view of the title compound, with the atomic labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Only one disorder component is shown.
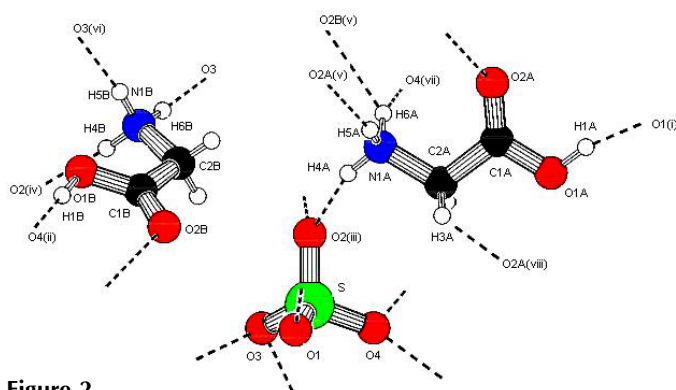

Figure 2
PLUTON (Spek, 1990) view of the title compound, showing the immediate hydrogen-bonding environment of the anion and cations.

these bonds involve atoms O2*A* and O2*B* of the glycinium cations (*A* and *B*) as donors and O atoms of sulfate anions as acceptors; details of hydrogen bonds are given in Table 1. Atom N1*B* forms three hydrogen bonds with sulfate anions. Weak hydrogen bonds are observed in the glycine *A* layers, between N and O atoms, and between C and O atoms.

Experimental

Colorless single crystals of DGS were obtained by slow evaporation, at room temperature, of an equimolar solution of glycine and sulfuric acid.

Crystal data

$2C_2H_6NO_2{}^+ \cdot SO_4{}^{2-}$	Mo $K\alpha$ radiation
$M_r = 248.23$	Cell parameters from 12129
Orthorhombic, $Pbca$	reflections
$a = 8.9350\,(4)$ Å	$\theta = 3.6–26.4°$
$b = 10.2770\,(3)$ Å	$\mu = 0.35$ mm^{-1}
$c = 21.7640\,(3)$ Å	$T = 293\,(2)$ K
$V = 1998.48\,(11)$ Å^3	Prism, colorless
$Z = 8$	$0.5 \times 0.4 \times 0.3$ mm
$D_x = 1.65$ Mg m^{-3}	

Data collection

Nonius KappaCCD diffractometer	$R_{int} = 0.092$
φ scans	$\theta_{max} = 26.4°$
Absorption correction: none	$h = -11 \rightarrow 11$
12129 measured reflections	$k = -12 \rightarrow 12$
1997 independent reflections	$l = -27 \rightarrow 25$
1844 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o{}^2) + (0.0331P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.034$	$+ 0.3591P]$
$wR(F^2) = 0.098$	where $P = (F_o{}^2 + 2F_c{}^2)/3$
$S = 1.10$	$(\Delta/\sigma)_{max} = 0.001$
1997 reflections	$\Delta\rho_{max} = 0.25$ e Å^{-3}
164 parameters	$\Delta\rho_{min} = -0.38$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	

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

$D—H\cdots A$	$D—H$	$H\cdots A$	$D\cdots A$	$D—H\cdots A$
O1A—H1$A\cdots$O1^i	0.82	1.77	2.567 (2)	164
O1B—H1$B\cdots$O4^{ii}	0.88 (3)	1.70 (3)	2.573 (2)	172 (3)
N1A—H4$A\cdots$O2^{iii}	0.89	1.92	2.762 (2)	156
N1B—H4$B\cdots$O2^{iv}	0.89	1.96	2.838 (2)	170
N1A—H5$A\cdots$O2A^v	0.89	2.13	2.958 (2)	154
N1B—H5$B\cdots$O3^{vi}	0.89	1.96	2.851 (2)	175
N1A—H6$A\cdots$O21B^v	0.89	2.48	3.018 (5)	119
N1A—H6$A\cdots$O22B^v	0.89	2.56	3.177 (6)	127
N1A—H6$A\cdots$O4^{vii}	0.89	2.03	2.805 (2)	145
N1B—H6$B\cdots$O3	0.89	1.93	2.813 (2)	170
C2A—H3$A\cdots$O2A^{viii}	0.97	2.51	3.264 (2)	134

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

In the initial refinement of the title compound, atom O2*B* showed high anisotropy of apparent thermal motion normal to the carboxyl plane. The final refinement was carried out with a model in which atoms O2*B* and C1*B* each have two alternative sites with equal occupancy, to simulate a disorder that occurs by a twist of the C1*B*—O2*B* arm. All H atoms (except H1*B*, which was refined isotropically) were then fixed at geometrically determined positions. Riding isotropic displacement parameters were used for all H atoms.

Data collection: *KappaCCD Reference Manual* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997) and *PLUTON* (Spek, 1990); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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supporting information

Diglycinium sulfate

Aouatef Cherouana, Nourredine Benali-Cherif, Lamia Bendjeddou and Hocine Merazig

S1. Comment

Many glycine salts and adducts exhibit interesting dielectric properties, the most well known compound of this family being TGS (triglycine sulfate), which orders ferroelectrically below 322 K (Matthias *et al.*, 1956). In addition of their main interest in the field of new materials chemistry (Siegel *et al.*, 1998; Baker *et al.*, 1992), hybrid compounds are of interest because of their electrical, magnetic and optical properties (Kagan *et al.*, 1999; Hill, 1998). The present compound is a result from a search of new organic–inorganic hybrid materials (Benali-Cherif, Cherouana *et al.*, 2002; Benali-Cherif, Abouimrane *et al.*, 2002; Benali-Cherif, Bendheif *et al.*, 2002; Benali-Cherif, Benguedouar *et al.*, 2002.). The asymmetric unit of DGS contains two monoprotaned glycine molecules ($C_2H_7NO_2^+$) and one anionic sulfate molecule (SO_4^{2-}). The mean bonds and angles in the SO_4^{2-} group are 1.472 Å and 109.465°, respectively, showing a normal tetrahedral geometry for the S atom. Interatomic distances in the glycine cations compare well with distances observed in diglycine sulfate monohydrate (Cano *et al.*, 1974). Although their carboxy skeletons are both planar, atom N1B is displaced from this plane by −0.170 (15) Å, whereas atom N1A is displaced by 0.075 (15) Å. The relevant torsion angles of the glycinium cations [O1A—C2A—C1A—N1A = 176.71 (15)° and N1B—C2B—C1B—O1B = 23.9 (7)°] indicate different conformations for the two glycinium cations in the asymmetric unit, *viz*. E for glycine A and Z for glycine B; this difference in conformation is not observed in diglycine selenate (Olejnik *et al.*, 1975). The crystal structure is built of two layers of glycine B ions and sulfate groups inserted between layers of glycine A along *b* axis. The layers are linked together by an intricate network of hydrogen-bond interactions. The strongest of these bonds involves atoms O2A and O2B of the glycinium cations (A and B) as donors and O atoms of sulfate anions as acceptors [O1A—H1A···O1 = 2.567 (2) Å and O1B—H1B···O4 = 2.573 (2) Å]. Atom N1B forms three mean hydrogen bonds with sulfate anion [N1B—H4B···O2 = 2.838 (2) Å, N1B—H5B···O3 = 2.851 (2) Å and N1B—H6B···O3 = 2.813 (2) Å]. Weak hydrogen bonds are observed in the glycine A layers, between N and O atoms [N1A—H5A···O2A = 2.958 (2) Å] and between C and O atoms [C2A—H3A···O2A = 3.264 (2) Å].

S2. Experimental

Colorless single crystals of DGS, were obtained by slow evaporation at room temperature of an equimolar solution of glycine and sulfuric acid.

S3. Refinement

In the initial refinement of the title compound, atom 2B showed high anisotropy of apparent thermal motion normal to the carboxyl plane. Final refinement was carried out with a model in which atoms O2B and C1B had a site-occupation factor of 0.5 (C1B/C11B and O2B/O21B), to simulate a dynamic disorder that occurs by a twist of the C1B—O2B arm. All H atoms were then fixed at localized positions. Riding isotropic displacement parameters were used for all H atoms.
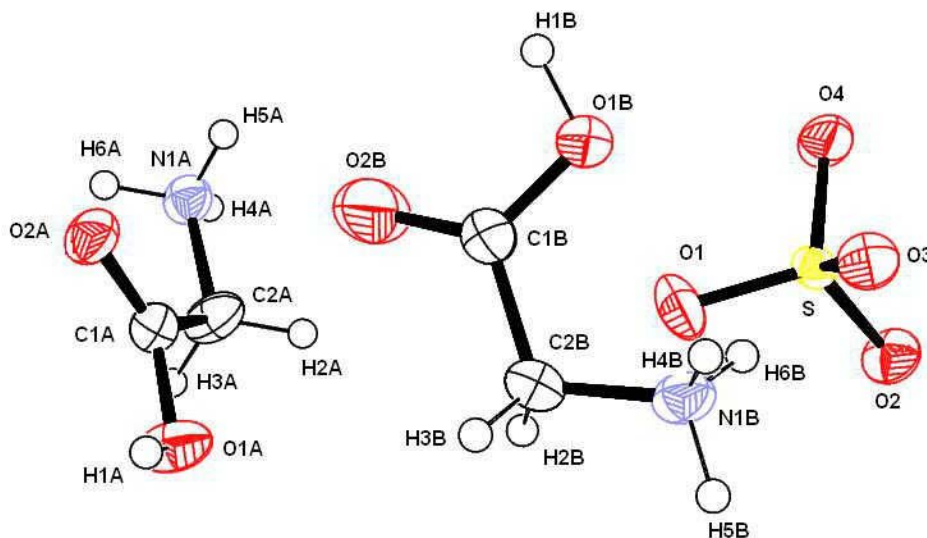
Figure 1

ORTEP-3 (Farrugia, 1997) view of the title compound with the atomic labelling scheme. Displacement are drawn at the 50% probability level.
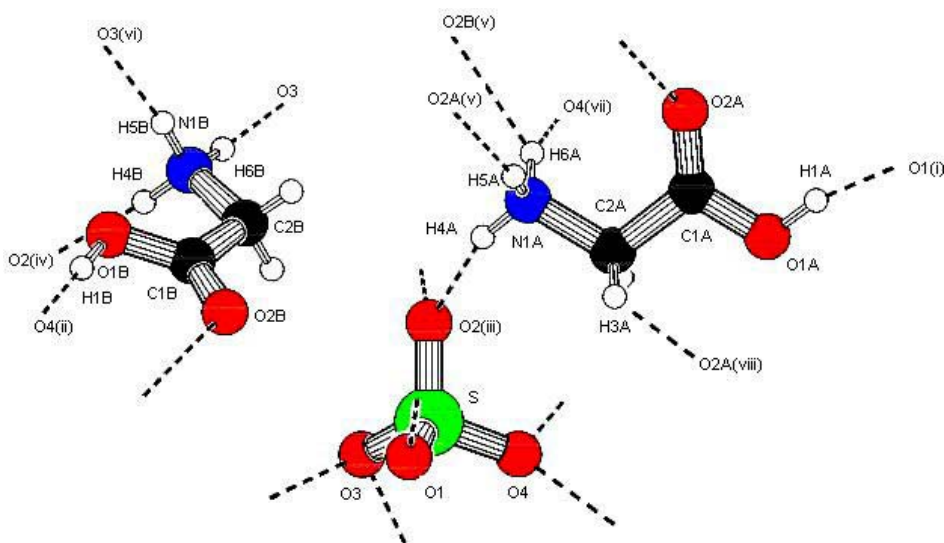
Figure 2

PLUTON (Spek, 1990) view of the title compound, showing the immediate hydrogen-bonded surrounding of the anion and cations.

(DGS)

Crystal data

$2C_2H_6NO_2{}^+ \cdot SO_4{}^{2-}$

$M_r = 248.23$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 8.9350\ (4)$ Å

$b = 10.2770\ (3)$ Å

$c = 21.7640\ (3)$ Å

$V = 1998.48\ (11)$ Å^3

$Z = 8$

$F(000) = 1040$

$D_x = 1.65$ Mg m^{-3}

Mo *Kα* radiation, $\lambda = 0.71073$ Å

Cell parameters from 12129 reflections

$\theta = 3.6$–$26.4°$

$\mu = 0.35$ mm^{-1}	Prism, colorless
$T = 293$ K	$0.5 \times 0.4 \times 0.3$ mm

Data collection

Nonius KappaCCD diffractometer	1844 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.092$
Graphite monochromator	$\theta_{max} = 26.4°$, $\theta_{min} = 3.2°$
φ scans	$h = -11 \rightarrow 11$
12129 measured reflections	$k = -12 \rightarrow 12$
1997 independent reflections	$l = -27 \rightarrow 25$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.034$	Hydrogen site location: difference Fourier map
$wR(F^2) = 0.098$	H atoms treated by a mixture of independent
$S = 1.10$	and constrained refinement
1997 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0331P)^2 + 0.3591P]$
164 parameters	where $P = (F_o^2 + 2F_c^2)/3$
24 restraints	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant	$\Delta\rho_{max} = 0.25$ e Å^{-3}
direct methods	$\Delta\rho_{min} = -0.38$ 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_{iso}*/U_{eq}	Occ. (<1)
S	−0.10690 (4)	0.55788 (4)	0.663471 (17)	0.02355 (15)	
O2A	0.21628 (14)	−0.03292 (11)	0.51481 (6)	0.0326 (3)	
O1	−0.00956 (16)	0.48644 (12)	0.62094 (6)	0.0415 (3)	
O3	−0.08780 (14)	0.50816 (11)	0.72625 (6)	0.0333 (3)	
O1A	0.38658 (14)	0.12148 (13)	0.53549 (7)	0.0409 (3)	
H1A	0.4201	0.0666	0.5591	0.061*	
O2	−0.07143 (15)	0.69813 (12)	0.66235 (6)	0.0357 (3)	
O4	−0.26467 (14)	0.53943 (13)	0.64542 (7)	0.0414 (3)	
N1A	0.07072 (16)	0.12375 (13)	0.43269 (7)	0.0297 (3)	
H6A	0.0957	0.0487	0.4150	0.045*	
H4A	0.0430	0.1804	0.4039	0.045*	
H5A	−0.0048	0.1107	0.4586	0.045*	
N1B	0.16743 (16)	0.34925 (14)	0.73588 (7)	0.0321 (3)	
H6B	0.0914	0.4030	0.7285	0.048*	
H5B	0.2471	0.3951	0.7474	0.048*	

H4B	0.1423	0.2941	0.7657	0.048*	
C2B	0.2030 (2)	0.2758 (2)	0.67961 (10)	0.0431 (5)	
H3B	0.2964	0.2293	0.6855	0.052*	
H2B	0.2165	0.3361	0.6458	0.052*	
C1AB	0.0806 (9)	0.1794 (8)	0.6633 (4)	0.034 (2)	0.50
O21B	0.1050 (6)	0.0918 (5)	0.6278 (3)	0.0689 (16)	0.50
O1B	−0.05002 (14)	0.21073 (12)	0.68600 (6)	0.0338 (3)	
C1BB	0.0690 (9)	0.2089 (8)	0.6509 (4)	0.0335 (19)	0.50
O22B	0.0744 (6)	0.1476 (6)	0.6040 (2)	0.0689 (15)	0.50
C1A	0.26723 (18)	0.07554 (15)	0.50827 (7)	0.0274 (3)	
C2A	0.1997 (2)	0.17554 (16)	0.46640 (8)	0.0352 (4)	
H2A	0.2748	0.2048	0.4373	0.042*	
H3A	0.1682	0.2501	0.4905	0.042*	
H1B	−0.119 (3)	0.158 (3)	0.6712 (13)	0.067 (8)*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0196 (2)	0.0202 (2)	0.0309 (2)	0.00172 (13)	0.00215 (14)	0.00150 (13)
O2A	0.0360 (7)	0.0215 (5)	0.0402 (6)	−0.0011 (5)	−0.0020 (5)	0.0008 (5)
O1	0.0424 (7)	0.0317 (6)	0.0505 (8)	0.0098 (6)	0.0231 (6)	0.0052 (5)
O3	0.0367 (7)	0.0295 (6)	0.0338 (6)	−0.0024 (5)	−0.0003 (5)	0.0058 (5)
O1A	0.0378 (7)	0.0370 (7)	0.0479 (8)	−0.0103 (5)	−0.0159 (6)	0.0112 (6)
O2	0.0425 (7)	0.0210 (6)	0.0437 (7)	−0.0031 (5)	0.0003 (5)	0.0053 (5)
O4	0.0243 (6)	0.0425 (7)	0.0575 (8)	0.0043 (5)	−0.0109 (6)	−0.0169 (6)
N1A	0.0306 (7)	0.0241 (6)	0.0344 (7)	0.0017 (5)	−0.0032 (6)	0.0016 (5)
N1B	0.0244 (7)	0.0291 (7)	0.0427 (8)	−0.0014 (6)	−0.0085 (6)	−0.0009 (6)
C2B	0.0225 (8)	0.0464 (11)	0.0603 (12)	−0.0021 (7)	0.0043 (8)	−0.0162 (9)
C1AB	0.033 (3)	0.034 (4)	0.035 (3)	−0.006 (2)	0.006 (2)	−0.005 (3)
O21B	0.045 (3)	0.081 (4)	0.081 (4)	−0.013 (2)	0.019 (2)	−0.052 (3)
O1B	0.0255 (6)	0.0334 (6)	0.0426 (7)	−0.0030 (5)	0.0015 (5)	−0.0071 (5)
C1BB	0.030 (2)	0.030 (3)	0.040 (4)	0.000 (2)	0.006 (2)	−0.007 (3)
O22B	0.047 (3)	0.088 (4)	0.072 (3)	−0.017 (3)	0.022 (2)	−0.048 (3)
C1A	0.0276 (8)	0.0250 (7)	0.0296 (7)	−0.0003 (6)	0.0008 (6)	−0.0010 (6)
C2A	0.0399 (9)	0.0246 (8)	0.0411 (9)	−0.0057 (7)	−0.0111 (8)	0.0053 (7)

Geometric parameters (Å, º)

S—O1	1.4670 (12)	C2B—C1BB	1.515 (8)
S—O3	1.4686 (12)	C2B—C1AB	1.517 (8)
S—O4	1.4756 (13)	C1AB—O21B	1.207 (8)
S—O2	1.4761 (12)	C1AB—O1B	1.307 (8)
O2A—C1A	1.212 (2)	O1B—C1BB	1.310 (8)
O1A—C1A	1.308 (2)	C1BB—O22B	1.201 (8)
N1A—C2A	1.466 (2)	C1A—C2A	1.500 (2)
N1B—C2B	1.474 (2)		
O1—S—O3	110.12 (7)	O21B—C1AB—C2B	120.4 (7)

O1—S—O4	109.52 (8)	O1B—C1AB—C2B	113.3 (6)
O3—S—O4	108.30 (8)	C1AB—O1B—C1BB	18.4 (6)
O1—S—O2	110.55 (8)	O22B—C1BB—O1B	122.4 (7)
O3—S—O2	109.28 (7)	O22B—C1BB—C2B	123.9 (7)
O4—S—O2	109.04 (8)	O1B—C1BB—C2B	113.3 (6)
N1B—C2B—C1BB	113.9 (3)	O2A—C1A—O1A	125.78 (15)
N1B—C2B—C1AB	111.9 (3)	O2A—C1A—C2A	123.36 (15)
C1BB—C2B—C1AB	15.9 (5)	O1A—C1A—C2A	110.85 (14)
O21B—C1AB—O1B	125.9 (7)	N1A—C2A—C1A	111.81 (13)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*A*—H1*A*···O1[i]	0.82	1.77	2.567 (2)	164
O1*B*—H1*B*···O4[ii]	0.88 (3)	1.70 (3)	2.573 (2)	172 (3)
N1*A*—H4*A*···O2[iii]	0.89	1.92	2.762 (2)	156
N1*B*—H4*B*···O2[iv]	0.89	1.96	2.838 (2)	170
N1*A*—H5*A*···O2*A*[v]	0.89	2.13	2.958 (2)	154
N1*B*—H5*B*···O3[vi]	0.89	1.96	2.851 (2)	175
N1*A*—H6*A*···O21*B*[v]	0.89	2.48	3.018 (5)	119
N1*A*—H6*A*···O22*B*[v]	0.89	2.56	3.177 (6)	127
N1*A*—H6*A*···O4[vii]	0.89	2.03	2.805 (2)	145
N1*B*—H6*B*···O3	0.89	1.93	2.813 (2)	170
C2*A*—H3*A*···O2*A*[viii]	0.97	2.51	3.264 (2)	134

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