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Bis[diamino(ethoxycarbonylamino)-methylium] sulfate

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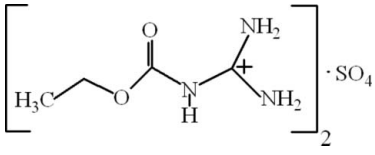
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.045; wR factor = 0.108; data-to-parameter ratio = 14.9.

In the molecule of the title compound, $2C_4H_{10}N_3O_2^+\cdot SO_4^-$, the cations are planar (r.m.s. deviations = 0.0144 and 0.0236 Å) and oriented at a dihedral angle of 62.30 (4)°. Intramolecular N—H···O hydrogen bonds result in the formation of two planar six-membered rings. The cations are linked to the sulfate ion through intermolecular C—H···O and N—H···O hydrogen bonds, forming an $R_2^2(8)$ ring motif. In the crystal structure, intermolecular N—H···O and C—H···O hydrogen bonds link the molecules into a three-dimensional network.

Related literature

For related structures, see: Brauer & Kottsieper (2003); Curtis & Pasternak (1955). For bond-length data, see: Allen *et al.* (1987). For ring motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$2C_4H_{10}N_3O_2^+\cdot SO_4^{2-}$
M_r = 360.36
Monoclinic, $P2_1/c$
a = 9.3021 (12) Å
b = 11.0081 (11) Å
c = 17.1063 (13) Å
β = 100.980 (3)°
V = 1719.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 296 K
0.24 × 0.18 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.946, T_{max} = 0.967
3481 measured reflections
3481 independent reflections
2124 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
3 standard reflections
frequency: 120 min
intensity decay: 1.7%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.108
S = 1.03
3481 reflections
233 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.95 (3)	1.89 (3)	2.837 (3)	175 (3)
N1—H1B···O2^{ii}	0.84 (3)	1.96 (3)	2.805 (3)	177 (3)
N2—H2A···O5	0.82 (3)	2.10 (3)	2.712 (3)	131 (3)
N2—H2A···O7^{iii}	0.82 (3)	2.27 (3)	2.975 (3)	144 (3)
N2—H2B···O1^{ii}	0.90 (3)	1.95 (3)	2.854 (3)	174 (3)
N4—H4D···O3^{i}	0.91 (3)	2.00 (3)	2.841 (3)	153 (2)
N4—H4E···O2	0.91 (3)	1.90 (3)	2.813 (3)	176 (3)
N3—H5···O4^{i}	0.86	1.94	2.769 (3)	163
N5—H5A···O7	0.81 (3)	2.14 (3)	2.730 (3)	130 (3)
N5—H5A···O5^{iv}	0.81 (3)	2.32 (3)	3.031 (3)	147 (3)
N5—H5B···O4	0.93 (3)	1.97 (3)	2.898 (3)	177 (3)
N6—H6···O3^{i}	0.86	1.95	2.752 (3)	155
C3—H3A···O3	0.97	2.58	3.368 (4)	138
C7—H7B···O2^{v}	0.97	2.55	3.483 (3)	162

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2641).

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supporting information

Acta Cryst. (2009). E**65**, o785 [doi:10.1107/S160053680900912X]

Bis[diamino(ethoxycarbonylamino)methylium] sulfate

M. Nawaz Tahir, Christy Muir, Muhammad Danish, Muhammad Ilyas Tariq and Dinçer Ülkü

S1. Comment

As part of our ongoing studies, we report herein the crystal structure of the title compound, (I).

The crystal structures of 1-carbamoylguanidinium methylphosphonate monohydrate, (II) (Brauer & Kottsieper, 2003) and methylguanidinium nitrate, (III) (Curtis & Pasternak, 1955) have been reported. In the molecule of the title compound (Fig. 1), the bond lengths (Allen *et al.*, 1987) and angles of the diamino(ethoxycarbonylamino)methylium (DEAM) moieties are within normal ranges. DEAM moieties (N1–N3/O5/O6/C1–C4) and (N4–N6/O7/O8/C5–C8) are planar with maximum deviations of 0.043 (2) and -0.322 (3) Å for N2 and N4 atoms, respectively, in which they are oriented at a dihedral angle of 62.30 (4)°. The intramolecular N—H···O hydrogen bonds result in the formations of two planar six-membered rings: A (O5/N2/N3/C1/C2/H2A) and B (O7/N5/N6/C5/C6/H5A). The dihedral angle between them is A/B = 60.38 (3)°. The DEAM moieties are linked to the SO_4 ion through the intramolecular C—H···O and N—H···O hydrogen bonds (Table 1), forming a $R_2^2(8)$ ring motif (Bernstein *et al.*, 1995).

In the crystal structure, intermolecular N—H···O and C—H···O hydrogen bonds (Table 1) link the molecules into a three dimensional network (Fig. 2), in which they may be effective in the stabilization of the structure.

S2. Experimental

For the preparation of the title compound, 1-cyanoguanidine (2.1 g, 0.025 mol) was dissolved in water (50 ml), and then a few drops of H_2SO_4 were added. The resulting mixture was refluxed for 2–3 h, and cooled to room temperature. The excess of ethanol was added, and then refluxed for 2–3 h. It was filtered through alumina. The filtrate was concentrated under reduced pressure and kept for crystallization. Recrystallization was carried out from ethanol/hexane (9:1) mixture in 5 d.

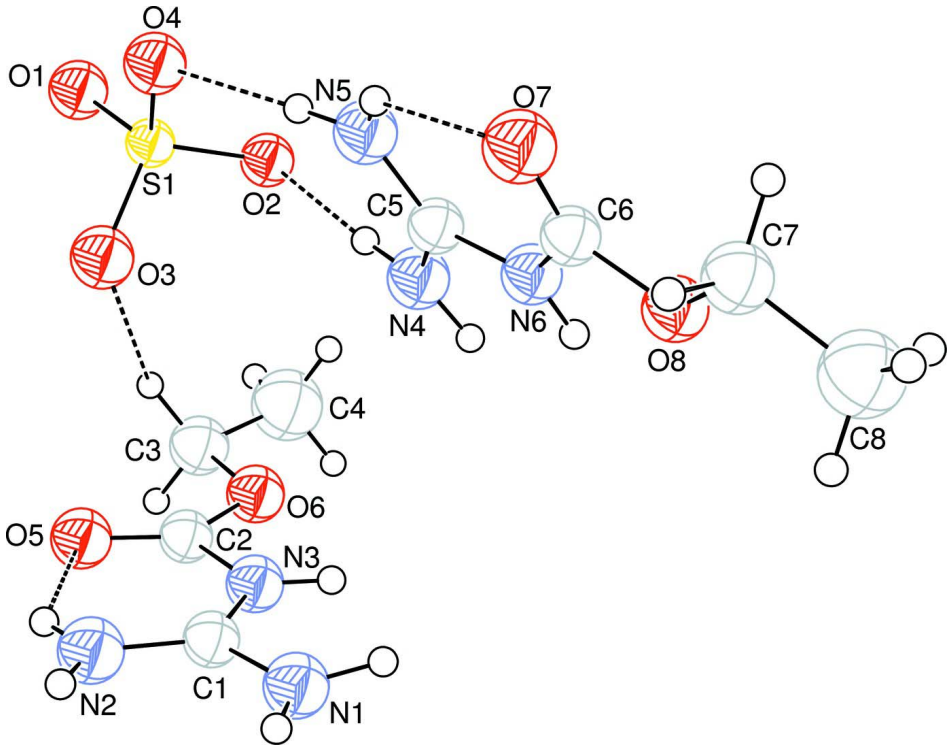

Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Hydrogen bonds are shown as dashed lines.

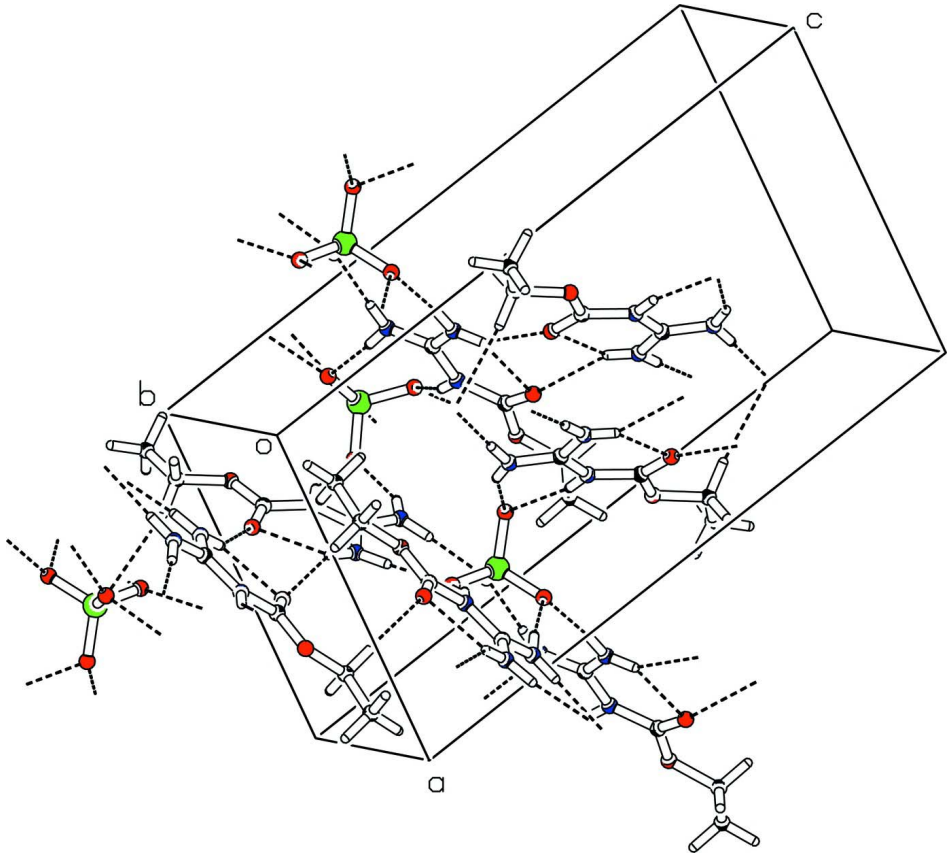

Figure 2

A partial packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

Bis[diamino(ethoxycarbonylamino)methylium] sulfate

Crystal data

$2C_4H_{10}N_3O_2^+\cdot SO_4^{2-}$
$M_r = 360.36$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.3021\ (12)$ Å
$b = 11.0081\ (11)$ Å
$c = 17.1063\ (13)$ Å
$\beta = 100.980\ (3)°$
$V = 1719.6\ (3)$ Å^3
$Z = 4$
$F(000) = 760$
$D_x = 1.392$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10.0–18.2°$
$\mu = 0.24$ mm^{-1}
$T = 296$ K
Prism, colourless
0.24 × 0.18 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.946$, $T_{max} = 0.967$
3481 measured reflections
3481 independent reflections
2124 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 26.3°$, $\theta_{min} = 2.2°$
$h = 0\rightarrow11$
$k = 0\rightarrow13$
$l = -21\rightarrow20$
3 standard reflections every 120 min
intensity decay: 1.7%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.108$
$S = 1.03$
3481 reflections
233 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
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0481P)^2 + 0.1093P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0037 (9)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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}
S1	0.74538 (6)	0.22561 (5)	0.25630 (4)	0.0324 (2)
O1	0.87642 (18)	0.15672 (16)	0.29258 (11)	0.0494 (6)
O2	0.78056 (17)	0.35534 (15)	0.25361 (10)	0.0398 (6)
O3	0.6301 (2)	0.20989 (17)	0.30259 (11)	0.0536 (7)
O4	0.6925 (2)	0.18149 (16)	0.17468 (10)	0.0492 (7)
O5	0.43306 (19)	0.28294 (17)	0.43071 (11)	0.0499 (6)
O6	0.53979 (17)	0.45797 (16)	0.40549 (11)	0.0471 (6)
O7	0.2706 (2)	0.42933 (17)	−0.03291 (11)	0.0560 (7)
O8	0.17269 (19)	0.60725 (16)	−0.00697 (10)	0.0460 (6)
N1	0.0671 (3)	0.4461 (2)	0.29228 (14)	0.0499 (8)
N2	0.1472 (3)	0.2719 (2)	0.35872 (15)	0.0529 (9)
N3	0.3058 (2)	0.43420 (19)	0.35471 (12)	0.0422 (7)
N4	0.5287 (3)	0.4909 (2)	0.19080 (14)	0.0500 (8)
N5	0.4929 (3)	0.3569 (2)	0.08613 (14)	0.0480 (8)
N6	0.3530 (2)	0.53306 (19)	0.08234 (12)	0.0426 (7)
C1	0.1711 (3)	0.3811 (2)	0.33493 (15)	0.0382 (8)
C2	0.4290 (3)	0.3811 (2)	0.40006 (15)	0.0381 (8)
C3	0.6785 (3)	0.4149 (3)	0.45146 (17)	0.0532 (10)
C4	0.7871 (3)	0.5128 (3)	0.4471 (2)	0.0807 (15)
C5	0.4611 (3)	0.4578 (2)	0.11953 (15)	0.0376 (8)
C6	0.2642 (3)	0.5151 (3)	0.00936 (15)	0.0387 (8)
C7	0.0712 (3)	0.6005 (3)	−0.08355 (15)	0.0502 (10)
C8	−0.0188 (4)	0.7126 (3)	−0.0906 (2)	0.0738 (12)
H1A	0.090 (3)	0.518 (3)	0.2665 (16)	0.0599*

H1B	−0.020 (3)	0.421 (3)	0.2797 (17)	0.0599*
H2A	0.215 (3)	0.234 (3)	0.3859 (18)	0.0634*
H2B	0.059 (3)	0.237 (3)	0.3410 (17)	0.0634*
H3A	0.70789	0.34004	0.42905	0.0638*
H3B	0.67011	0.40013	0.50630	0.0638*
H4A	0.88070	0.48943	0.47744	0.0965*
H4B	0.75542	0.58660	0.46846	0.0965*
H4C	0.79518	0.52545	0.39258	0.0965*
H4D	0.499 (3)	0.563 (3)	0.2083 (16)	0.0600*
H4E	0.608 (3)	0.445 (3)	0.2123 (16)	0.0600*
H5	0.31494	0.50670	0.33757	0.0505*
H5A	0.446 (3)	0.337 (3)	0.0435 (17)	0.0576*
H5B	0.560 (3)	0.303 (3)	0.1145 (16)	0.0576*
H6	0.33899	0.59866	0.10715	0.0511*
H7A	0.00927	0.52921	−0.08540	0.0602*
H7B	0.12481	0.59560	−0.12684	0.0602*
H8A	−0.09353	0.70817	−0.13771	0.0883*
H8B	0.04250	0.78183	−0.09397	0.0883*
H8C	−0.06354	0.72035	−0.04471	0.0883*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0322 (3)	0.0241 (3)	0.0383 (4)	−0.0023 (3)	−0.0001 (3)	0.0012 (3)
O1	0.0432 (11)	0.0334 (10)	0.0616 (12)	0.0064 (9)	−0.0152 (9)	−0.0021 (9)
O2	0.0347 (9)	0.0263 (9)	0.0566 (11)	−0.0042 (7)	0.0041 (8)	0.0042 (8)
O3	0.0547 (12)	0.0461 (12)	0.0647 (12)	−0.0088 (10)	0.0234 (10)	0.0106 (10)
O4	0.0594 (12)	0.0378 (11)	0.0425 (11)	0.0086 (9)	−0.0106 (9)	−0.0060 (8)
O5	0.0432 (10)	0.0396 (11)	0.0625 (12)	−0.0056 (9)	−0.0011 (9)	0.0182 (10)
O6	0.0315 (9)	0.0409 (11)	0.0639 (12)	−0.0073 (8)	−0.0032 (8)	0.0060 (9)
O7	0.0609 (12)	0.0462 (12)	0.0535 (12)	0.0160 (10)	−0.0080 (9)	−0.0187 (10)
O8	0.0512 (11)	0.0440 (11)	0.0394 (10)	0.0178 (9)	0.0002 (8)	−0.0017 (9)
N1	0.0359 (12)	0.0444 (15)	0.0620 (16)	−0.0101 (12)	−0.0097 (12)	0.0166 (12)
N2	0.0430 (14)	0.0397 (15)	0.0673 (16)	−0.0156 (12)	−0.0113 (12)	0.0165 (12)
N3	0.0344 (11)	0.0336 (13)	0.0537 (13)	−0.0085 (10)	−0.0036 (10)	0.0124 (10)
N4	0.0540 (15)	0.0426 (15)	0.0466 (14)	0.0216 (12)	−0.0074 (11)	−0.0097 (12)
N5	0.0548 (15)	0.0379 (14)	0.0451 (14)	0.0159 (12)	−0.0062 (11)	−0.0088 (12)
N6	0.0498 (13)	0.0340 (12)	0.0408 (12)	0.0139 (11)	0.0006 (10)	−0.0078 (10)
C1	0.0365 (14)	0.0356 (16)	0.0394 (14)	−0.0084 (12)	−0.0004 (11)	0.0031 (12)
C2	0.0367 (14)	0.0365 (16)	0.0394 (14)	−0.0071 (12)	0.0033 (11)	0.0010 (12)
C3	0.0355 (15)	0.0536 (19)	0.0644 (19)	−0.0006 (14)	−0.0058 (13)	−0.0074 (15)
C4	0.0395 (17)	0.093 (3)	0.105 (3)	−0.0204 (18)	0.0024 (17)	−0.012 (2)
C5	0.0391 (14)	0.0342 (15)	0.0388 (15)	0.0066 (12)	0.0055 (12)	−0.0001 (12)
C6	0.0396 (14)	0.0345 (15)	0.0417 (15)	0.0051 (12)	0.0069 (12)	−0.0004 (13)
C7	0.0500 (16)	0.0579 (19)	0.0384 (15)	0.0098 (15)	−0.0022 (12)	0.0020 (14)
C8	0.070 (2)	0.075 (2)	0.070 (2)	0.032 (2)	−0.0028 (17)	0.0115 (19)

Geometric parameters (Å, º)

S1—O3	1.460 (2)	N5—C5	1.308 (3)
S1—O4	1.4716 (18)	N6—C5	1.363 (3)
S1—O1	1.4690 (19)	N6—C6	1.374 (3)
S1—O2	1.4678 (17)	N4—H4E	0.91 (3)
O5—C2	1.199 (3)	N4—H4D	0.91 (3)
O6—C3	1.457 (3)	N5—H5B	0.93 (3)
O6—C2	1.323 (3)	N5—H5A	0.81 (3)
O7—C6	1.198 (4)	N6—H6	0.8600
O8—C6	1.320 (4)	C3—C4	1.489 (4)
O8—C7	1.464 (3)	C3—H3A	0.9700
N1—C1	1.308 (4)	C3—H3B	0.9700
N2—C1	1.302 (3)	C4—H4C	0.9600
N3—C2	1.385 (3)	C4—H4B	0.9600
N3—C1	1.366 (3)	C4—H4A	0.9600
N1—H1A	0.95 (3)	C7—C8	1.483 (5)
N1—H1B	0.84 (3)	C7—H7A	0.9700
N2—H2A	0.82 (3)	C7—H7B	0.9700
N2—H2B	0.90 (3)	C8—H8B	0.9600
N3—H5	0.8600	C8—H8C	0.9600
N4—C5	1.312 (3)	C8—H8A	0.9600
S1···H2B^{i}	3.00 (3)	N4···O2	2.813 (3)
S1···H4E	2.77 (3)	N5···O4	2.898 (3)
S1···H5B	2.83 (3)	N5···O7	2.730 (3)
S1···H1A^{ii}	2.82 (3)	N5···C6^{v}	3.341 (4)
S1···H4D^{ii}	3.04 (3)	N5···O5^{iv}	3.031 (3)
S1···H5^{ii}	2.8900	N6···O3^{vii}	2.752 (3)
S1···H6^{ii}	2.9500	C2···O3	3.320 (3)
S1···H1B^{i}	3.04 (3)	C3···O3	3.368 (4)
O1···N2^{i}	2.854 (3)	C5···O3^{vii}	3.260 (3)
O1···N1^{ii}	2.837 (3)	C5···O7^{v}	3.373 (3)
O2···N1^{i}	2.805 (3)	C6···N5^{v}	3.341 (4)
O2···N4	2.813 (3)	C2···H4B^{viii}	3.1000
O3···C2	3.320 (3)	C2···H2A	2.54 (3)
O3···N4^{ii}	2.841 (3)	C6···H5A	2.58 (3)
O3···C5^{ii}	3.260 (3)	H1A···S1^{vii}	2.82 (3)
O3···C3	3.368 (4)	H1A···O1^{vii}	1.89 (3)
O3···O5	3.216 (3)	H1A···H5	2.2100
O3···N6^{ii}	2.752 (3)	H1A···O4^{vii}	2.75 (3)
O4···N5	2.898 (3)	H1B···S1^{vi}	3.04 (3)
O4···N3^{ii}	2.769 (3)	H1B···H2B	2.33 (5)
O5···O7^{iii}	2.914 (3)	H1B···O2^{vi}	1.96 (3)
O5···O3	3.216 (3)	H2A···O7^{iii}	2.27 (3)
O5···N2	2.712 (3)	H2A···C2	2.54 (3)
O5···N5^{iii}	3.031 (3)	H2A···O5	2.10 (3)
O7···O5^{iv}	2.914 (3)	H2B···O1^{vi}	1.95 (3)

O7···N5	2.730 (3)	H2B···H1B	2.33 (5)
O7···N2^{iv}	2.975 (3)	H2B···S1^{vi}	3.00 (3)
O7···C5^{v}	3.373 (3)	H3A···O5	2.6400
O1···H2B^{i}	1.95 (3)	H3A···O3	2.5800
O1···H1A^{ii}	1.89 (3)	H3B···O5	2.6700
O2···H1B^{i}	1.96 (3)	H4A···H4A^{ix}	2.2200
O2···H5B	2.89 (3)	H4B···C2^{viii}	3.1000
O2···H4E	1.90 (3)	H4D···S1^{vii}	3.04 (3)
O2···H7B^{v}	2.5500	H4D···H6	2.0900
O3···H3A	2.5800	H4D···O3^{vii}	2.00 (3)
O3···H4D^{ii}	2.00 (3)	H4E···S1	2.77 (3)
O3···H6^{ii}	1.9500	H4E···H5B	2.27 (4)
O4···H5B	1.97 (3)	H4E···O2	1.90 (3)
O4···H1A^{ii}	2.75 (3)	H5···H1A	2.2100
O4···H5^{ii}	1.9400	H5···S1^{vii}	2.8900
O5···H3A	2.6400	H5···O4^{vii}	1.9400
O5···H3B	2.6700	H5A···C6	2.58 (3)
O5···H5A^{iii}	2.32 (3)	H5A···O5^{iv}	2.32 (3)
O5···H2A	2.10 (3)	H5A···O7	2.14 (3)
O7···H7B	2.6300	H5B···O2	2.89 (3)
O7···H7A	2.6600	H5B···O4	1.97 (3)
O7···H5A	2.14 (3)	H5B···S1	2.83 (3)
O7···H2A^{iv}	2.27 (3)	H5B···H4E	2.27 (4)
N1···O2^{vi}	2.805 (3)	H6···S1^{vii}	2.9500
N1···O1^{vii}	2.837 (3)	H6···O3^{vii}	1.9500
N2···O1^{vi}	2.854 (3)	H6···H4D	2.0900
N2···O5	2.712 (3)	H7A···O7	2.6600
N2···O7^{iii}	2.975 (3)	H7B···O7	2.6300
N3···O4^{vii}	2.769 (3)	H7B···O2^{v}	2.5500
N4···O3^{vii}	2.841 (3)		

O3—S1—O4	109.16 (11)	O5—C2—N3	125.4 (2)
O1—S1—O3	110.23 (11)	O6—C3—C4	106.1 (2)
O1—S1—O4	109.33 (11)	H3A—C3—H3B	109.00
O1—S1—O2	110.09 (10)	O6—C3—H3B	111.00
O2—S1—O4	109.12 (10)	O6—C3—H3A	111.00
O2—S1—O3	108.88 (10)	C4—C3—H3A	111.00
C2—O6—C3	115.3 (2)	C4—C3—H3B	111.00
C6—O8—C7	115.5 (2)	C3—C4—H4C	109.00
C1—N3—C2	125.5 (2)	H4A—C4—H4B	109.00
C1—N1—H1A	120.5 (17)	H4A—C4—H4C	109.00
C1—N1—H1B	122 (2)	C3—C4—H4A	109.00
H1A—N1—H1B	116 (3)	C3—C4—H4B	109.00
C1—N2—H2A	119 (2)	H4B—C4—H4C	109.00
C1—N2—H2B	119 (2)	N4—C5—N5	122.2 (2)
H2A—N2—H2B	122 (3)	N4—C5—N6	116.4 (2)
C2—N3—H5	117.00	N5—C5—N6	121.3 (2)
C1—N3—H5	117.00	O7—C6—N6	124.9 (3)

C5—N6—C6	126.8 (2)	O7—C6—O8	125.6 (2)
C5—N4—H4E	115.4 (18)	O8—C6—N6	109.5 (2)
H4D—N4—H4E	129 (3)	O8—C7—C8	106.8 (2)
C5—N4—H4D	115.2 (17)	C8—C7—H7A	110.00
C5—N5—H5B	120.0 (18)	C8—C7—H7B	110.00
C5—N5—H5A	120 (2)	O8—C7—H7A	110.00
H5A—N5—H5B	120 (3)	O8—C7—H7B	110.00
C6—N6—H6	117.00	H7A—C7—H7B	109.00
C5—N6—H6	117.00	H8B—C8—H8C	109.00
N2—C1—N3	121.4 (2)	C7—C8—H8A	109.00
N1—C1—N3	116.8 (2)	C7—C8—H8B	109.00
N1—C1—N2	121.8 (3)	C7—C8—H8C	109.00
O6—C2—N3	108.65 (19)	H8A—C8—H8B	109.00
O5—C2—O6	125.9 (2)	H8A—C8—H8C	109.00
C3—O6—C2—O5	1.9 (4)	C2—N3—C1—N2	0.4 (4)
C3—O6—C2—N3	−179.8 (2)	C1—N3—C2—O5	−3.0 (4)
C2—O6—C3—C4	178.0 (2)	C1—N3—C2—O6	178.6 (2)
C6—O8—C7—C8	−179.0 (2)	C6—N6—C5—N4	−177.4 (3)
C7—O8—C6—O7	0.5 (4)	C6—N6—C5—N5	1.2 (4)
C7—O8—C6—N6	179.8 (2)	C5—N6—C6—O7	−0.7 (4)
C2—N3—C1—N1	179.2 (2)	C5—N6—C6—O8	180.0 (2)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{vii}	0.95 (3)	1.89 (3)	2.837 (3)	175 (3)
N1—H1*B*···O2^{vi}	0.84 (3)	1.96 (3)	2.805 (3)	177 (3)
N2—H2*A*···O5	0.82 (3)	2.10 (3)	2.712 (3)	131 (3)
N2—H2*A*···O7^{iii}	0.82 (3)	2.27 (3)	2.975 (3)	144 (3)
N2—H2*B*···O1^{vi}	0.90 (3)	1.95 (3)	2.854 (3)	174 (3)
N4—H4*D*···O3^{vii}	0.91 (3)	2.00 (3)	2.841 (3)	153 (2)
N4—H4*E*···O2	0.91 (3)	1.90 (3)	2.813 (3)	176 (3)
N3—H5···O4^{vii}	0.86	1.94	2.769 (3)	163
N5—H5*A*···O7	0.81 (3)	2.14 (3)	2.730 (3)	130 (3)
N5—H5*A*···O5^{iv}	0.81 (3)	2.32 (3)	3.031 (3)	147 (3)
N5—H5*B*···O4	0.93 (3)	1.97 (3)	2.898 (3)	177 (3)
N6—H6···O3^{vii}	0.86	1.95	2.752 (3)	155
C3—H3*A*···O3	0.97	2.58	3.368 (4)	138
C7—H7*B*···O2^{v}	0.97	2.55	3.483 (3)	162

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