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## Structure Reports

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## 2-Methyl-4-oxopentan-2-aminium 2-sulfamoylbenzoate

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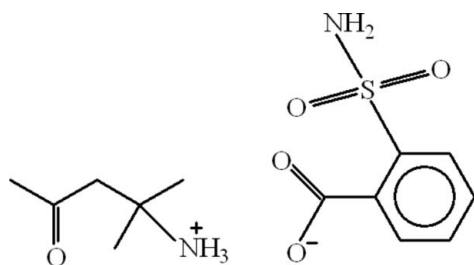
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 Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.037;  $wR$  factor = 0.110; data-to-parameter ratio = 20.6.

In the title salt,  $\text{C}_6\text{H}_{14}\text{NO}^+\cdot\text{C}_7\text{H}_6\text{NO}_4\text{S}^-$ , the 2-sulfamoylbenzoate anion has two intramolecular hydrogen bonds, forming a five membered  $\text{C}-\text{H}\cdots\text{O}$  and a seven-membered  $\text{N}-\text{H}\cdots\text{O}$  twisted ring with ring motifs  $S(5)$  and  $S(7)$ , respectively, while the 2-methyl-4-oxopentan-2-aminium cation also has a stabilizing intramolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bond with a twisted  $S(6)$  ring motif. The anions form inversion-related dimers with  $R_2^2(8)$  ring motifs through intermolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonding. The dimers and cations are further linked and stabilized through intermolecular  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{O}$  bonds, forming zigzag-shaped layers that extend along the crystallographic  $a$  axis.

### Related literature

For related structures, see: Akram *et al.* (2008); Schmidt *et al.* (1997); Siddiqui *et al.* (2007). For the definition of hydrogen-bond patterns used for graph-set analysis, see: Bernstein *et al.* (1995). For applications of aldol condensation, see: Afonso & Crespo (2005).



### Experimental

#### Crystal data

 $\text{C}_6\text{H}_{14}\text{NO}^+\cdot\text{C}_7\text{H}_6\text{NO}_4\text{S}^-$ 
 $M_r = 316.37$ 

 Orthorhombic,  $Pbca$ 
 $a = 21.1917$  (9) Å

 $b = 6.3897$  (2) Å

 $c = 23.3751$  (11) Å

 $V = 3165.2$  (2) Å<sup>3</sup>
 $Z = 8$ 

 Mo  $K\alpha$  radiation

 $\mu = 0.23$  mm<sup>-1</sup>
 $T = 296$  K

 $0.28 \times 0.10 \times 0.08$  mm

#### Data collection

Bruker Kappa APEXII CCD diffractometer

Absorption correction: multi-scan

 (*SADABS*; Bruker, 2005)

 $T_{\min} = 0.975$ ,  $T_{\max} = 0.983$ 

32870 measured reflections

4111 independent reflections

 2961 reflections with  $I > 2\sigma(I)$ 
 $R_{\text{int}} = 0.032$ 

#### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.037$ 
 $wR(F^2) = 0.110$ 
 $S = 1.04$ 

4111 reflections

200 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.32$  e Å<sup>-3</sup>
 $\Delta\rho_{\text{min}} = -0.29$  e Å<sup>-3</sup>
**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O4}^{\text{i}}$	0.80 (2)	2.21 (2)	3.008 (2)	172 (2)
$\text{N1}-\text{H1B}\cdots\text{O2}$	0.85 (2)	2.10 (2)	2.848 (2)	145.4 (18)
$\text{N2}-\text{H2A}\cdots\text{O1}$	0.89	1.94	2.8124 (18)	167
$\text{N2}-\text{H2B}\cdots\text{O2}^{\text{ii}}$	0.89	1.87	2.7622 (18)	176
$\text{N2}-\text{H2C}\cdots\text{O5}$	0.89	2.18	2.824 (2)	129
$\text{N2}-\text{H2C}\cdots\text{O1}^{\text{iii}}$	0.89	2.50	2.9878 (17)	115
$\text{C3}-\text{H3}\cdots\text{O4}$	0.93	2.47	2.870 (2)	106
$\text{C6}-\text{H6}\cdots\text{O2}^{\text{iii}}$	0.93	2.52	3.444 (2)	170

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINTE* (Bruker, 2007); data reduction: *SAINTE*; 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2187).

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## supporting information

*Acta Cryst.* (2009). E65, o1883 [doi:10.1107/S1600536809027007]

## 2-Methyl-4-oxopentan-2-aminium 2-sulfamoylbenzoate

Muhammad Rafique, Ghulam Hussain, Waseeq Ahmad Siddiqui and M. Nawaz Tahir

### S1. Comment

We reported the crystal structure of Tetraaquabis(2-sulfamoylbenzoato) manganese(II) (Akram *et al.*, 2008). In continuation of synthesizing metal complexes of *o*-sulfamoylbenzoic acid (Siddiqui *et al.*, 2007), the title compound (I), (Fig. 1) is prepared in a try of tin complex. The crystal structure of bis(2-Methyl-4-oxopent-2-yl)ammonium bis-(phthalato)-beryllium(I) (Schmidt *et al.*, 1997) contains the cation, 2-Methyl-4-oxopentan-2-aminium, of (I). The title compound is an example of aldol condensation which is routinely applied to prepare products used in the fields of biological sciences, industrial catalysis and green chemistry (Afonso & Crespo, 2005).

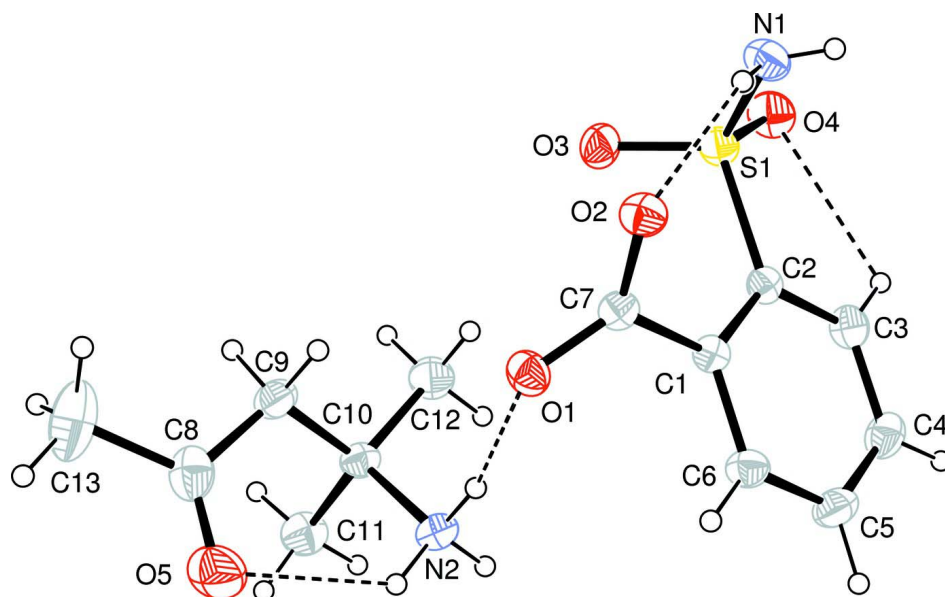
In the title compound, there are two moieties. In the anion, 2-sulfamoylbenzoate, two intramolecular H-bonds form five and seven membered twisted rings [S(5) and S(7)], while the cation, 2-methyl-4-oxopentan-2-aminium, has also an intramolecular H-bonding with a twisted ring S(6) (Bernstein *et al.*, 1995) (Fig. 1). The cations and anions are connected with each other through H-bonding of the N—H $\cdots$ O type. The 2-sulfamoylbenzoate form dimers with R<sub>2</sub><sup>2</sup>(8) ring motifs through intermolecular H-bonding of type N1—H1A $\cdots$ O4<sup>i</sup> (Table 1) [symmetry code: *i* = -*x*, -*y*, -*z* + 1]. The dimers are linked to 2-methyl-4-oxopentan-2-aminium moieties through intermolecular H-bondings, (Table 1, Fig. 2), forming zigzag shaped layers that extend along the crystallographic *a*-axis.

### S2. Experimental

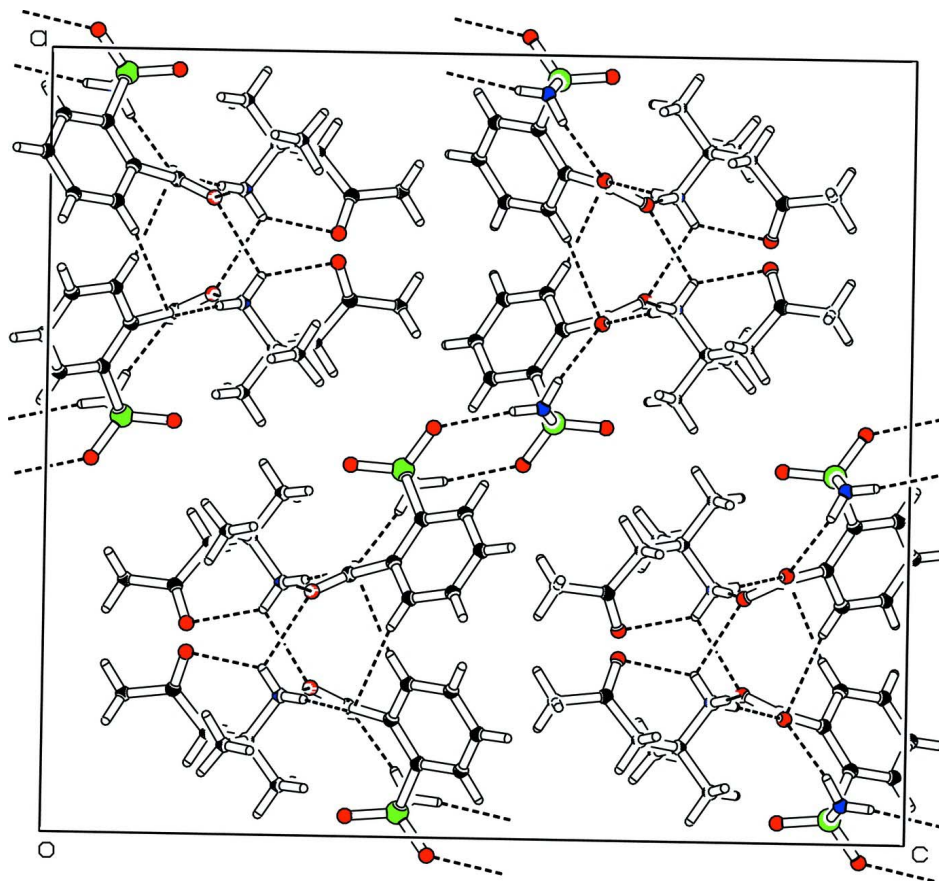
A suspension of (1.0 g, 4.97 mmol) *o*-sulfamoylbenzoic acid (Siddiqui *et al.*, 2007), tin(II) chloride dihydrate (0.561 g, 2.49 mmol) and sodium carbonate (0.3 g, 2.83 mmol) was subjected to reflux in a mixture of acetone, methanol, water (1:1:1) for 4 h. The volume of the reaction mixture was reduced to half on a rotary evaporator (11 torr) at room temperature and its pH was adjusted to 12, using aqueous ammonia solution and left over night at the same temperature. The white product was filtered, washed with cold distilled water and dried at room temperature. The product was recrystallized at 313 K from aqueous methanol to obtain colorless needle shaped crystals.

### S3. Refinement

The coordinates of H-atoms of NH<sub>2</sub> group were refined. The H-atoms were positioned geometrically, with N—H = 0.89 for NH<sub>3</sub>, C—H = 0.93, 0.96 and 0.97 Å for aryl, methyl and ethylenic H, respectively and constrained to ride on their parent atoms, with U<sub>iso</sub>(H) = xU<sub>eq</sub>(C, N), where x = 1.2 for all H atoms.

**Figure 1**

View of the title compound with the atom numbering scheme. The thermal ellipsoids are drawn at the 30% probability level. H-atoms are shown by small circles of arbitrary radii. Intramolecular H-bonds are shown by dotted lines.



**Figure 2**

The partial packing (*PLATON*; Spek, 2009) which shows that molecules form dimers of 2-sulfamoylbenzoate and connected to 2-methyl-4-oxopentan-2-aminium through intermolecular H-bondings.

### 2-Methyl-4-oxopentan-2-aminium 2-sulfamoylbenzoate

#### Crystal data

$C_6H_{14}NO^+ \cdot C_7H_6NO_4S^-$

$M_r = 316.37$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 21.1917(9) \text{ \AA}$

$b = 6.3897(2) \text{ \AA}$

$c = 23.3751(11) \text{ \AA}$

$V = 3165.2(2) \text{ \AA}^3$

$Z = 8$

$F(000) = 1344$

$D_x = 1.328 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 4111 reflections

$\theta = 2.6\text{--}28.8^\circ$

$\mu = 0.23 \text{ mm}^{-1}$

$T = 296 \text{ K}$

Needle, colorless

$0.28 \times 0.10 \times 0.08 \text{ mm}$

#### Data collection

Bruker Kappa APEXII CCD  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution:  $7.40 \text{ pixels mm}^{-1}$

$\omega$  scans

Absorption correction: multi-scan  
(*SADABS*; Bruker, 2005)

$T_{\min} = 0.975$ ,  $T_{\max} = 0.983$

32870 measured reflections

4111 independent reflections

2961 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.032$

$\theta_{\max} = 28.8^\circ$ ,  $\theta_{\min} = 2.6^\circ$   
 $h = -27 \rightarrow 28$

$k = -8 \rightarrow 5$   
 $l = -31 \rightarrow 30$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.037$   
 $wR(F^2) = 0.110$   
 $S = 1.04$   
 4111 reflections  
 200 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  
 $w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 1.1665P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.32 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.29 \text{ e } \text{\AA}^{-3}$

*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 esds 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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
O5	0.23168 (8)	0.6184 (3)	0.16582 (7)	0.0740 (6)
N2	0.17797 (6)	0.6721 (2)	0.27542 (6)	0.0367 (4)
C8	0.18387 (11)	0.5283 (3)	0.15179 (8)	0.0534 (6)
C9	0.12913 (9)	0.5023 (3)	0.19207 (8)	0.0451 (5)
C10	0.12064 (7)	0.6719 (3)	0.23730 (7)	0.0368 (5)
C11	0.11386 (9)	0.8877 (3)	0.21123 (9)	0.0504 (6)
C12	0.06400 (8)	0.6216 (3)	0.27479 (9)	0.0523 (6)
C13	0.17685 (15)	0.4323 (5)	0.09405 (10)	0.0902 (10)
S1	0.03137 (2)	0.12048 (7)	0.41313 (2)	0.0366 (1)
O1	0.18858 (5)	0.25236 (19)	0.31033 (5)	0.0426 (3)
O2	0.15851 (5)	-0.02972 (17)	0.35914 (5)	0.0413 (4)
O3	0.02610 (5)	0.1446 (2)	0.35274 (5)	0.0482 (4)
O4	-0.02263 (5)	0.1683 (2)	0.44799 (6)	0.0519 (4)
N1	0.04974 (8)	-0.1189 (2)	0.42641 (8)	0.0464 (5)
C1	0.15011 (7)	0.2984 (2)	0.40450 (6)	0.0307 (4)
C2	0.09432 (7)	0.2818 (2)	0.43663 (6)	0.0317 (4)
C3	0.08632 (8)	0.3954 (3)	0.48632 (7)	0.0408 (5)
C4	0.13337 (9)	0.5284 (3)	0.50512 (8)	0.0465 (6)
C5	0.18748 (9)	0.5514 (3)	0.47338 (8)	0.0473 (6)
C6	0.19563 (8)	0.4383 (3)	0.42347 (7)	0.0405 (5)
C7	0.16605 (7)	0.1638 (2)	0.35297 (7)	0.0324 (4)
H2A	0.18196	0.54745	0.29202	0.0441*
H2B	0.17365	0.77014	0.30218	0.0441*

H2C	0.21218	0.69889	0.25458	0.0441*
H9A	0.13377	0.36904	0.21148	0.0540*
H9B	0.09076	0.49517	0.16952	0.0540*
H11A	0.15160	0.92237	0.19057	0.0605*
H11B	0.10717	0.98862	0.24105	0.0605*
H11C	0.07852	0.88900	0.18554	0.0605*
H12A	0.06955	0.48618	0.29186	0.0628*
H12B	0.02641	0.62158	0.25188	0.0628*
H12C	0.06029	0.72529	0.30432	0.0628*
H13A	0.21496	0.45282	0.07257	0.1083*
H13B	0.14222	0.49701	0.07436	0.1083*
H13C	0.16884	0.28511	0.09803	0.1083*
H1A	0.0454 (10)	-0.141 (3)	0.4599 (10)	0.0557*
H1B	0.0843 (11)	-0.148 (3)	0.4094 (9)	0.0557*
H3	0.04917	0.38246	0.50721	0.0490*
H4	0.12842	0.60205	0.53914	0.0558*
H5	0.21875	0.64337	0.48550	0.0568*
H6	0.23233	0.45630	0.40221	0.0486*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O5	0.0674 (10)	0.0762 (11)	0.0783 (11)	-0.0084 (8)	0.0259 (8)	-0.0127 (9)
N2	0.0321 (6)	0.0351 (7)	0.0430 (8)	-0.0012 (5)	-0.0019 (6)	-0.0067 (6)
C8	0.0666 (13)	0.0476 (10)	0.0461 (10)	0.0138 (10)	0.0033 (9)	-0.0028 (9)
C9	0.0520 (10)	0.0389 (8)	0.0443 (10)	-0.0064 (8)	-0.0092 (8)	-0.0041 (7)
C10	0.0326 (8)	0.0361 (8)	0.0417 (9)	-0.0027 (6)	-0.0045 (7)	-0.0009 (7)
C11	0.0517 (10)	0.0403 (9)	0.0593 (12)	0.0047 (8)	-0.0053 (9)	0.0025 (9)
C12	0.0348 (9)	0.0671 (12)	0.0550 (11)	-0.0065 (8)	-0.0003 (8)	0.0020 (10)
C13	0.110 (2)	0.110 (2)	0.0506 (13)	0.0401 (18)	-0.0048 (13)	-0.0204 (14)
S1	0.0269 (2)	0.0441 (2)	0.0389 (2)	-0.0018 (2)	0.0009 (2)	0.0023 (2)
O1	0.0429 (6)	0.0449 (6)	0.0401 (6)	0.0007 (5)	0.0096 (5)	-0.0001 (5)
O2	0.0435 (6)	0.0334 (6)	0.0469 (7)	0.0033 (5)	0.0055 (5)	-0.0053 (5)
O3	0.0396 (6)	0.0637 (8)	0.0412 (7)	-0.0052 (6)	-0.0085 (5)	0.0042 (6)
O4	0.0308 (6)	0.0665 (8)	0.0583 (8)	0.0012 (5)	0.0095 (5)	0.0013 (7)
N1	0.0480 (8)	0.0423 (8)	0.0489 (9)	-0.0057 (7)	0.0092 (7)	0.0034 (7)
C1	0.0286 (7)	0.0301 (7)	0.0335 (8)	0.0032 (5)	-0.0002 (6)	0.0007 (6)
C2	0.0293 (7)	0.0327 (7)	0.0332 (8)	0.0033 (6)	-0.0012 (6)	0.0027 (6)
C3	0.0397 (8)	0.0444 (9)	0.0383 (9)	0.0057 (7)	0.0050 (7)	-0.0009 (7)
C4	0.0569 (11)	0.0444 (9)	0.0382 (9)	0.0048 (8)	-0.0033 (8)	-0.0103 (8)
C5	0.0493 (10)	0.0416 (9)	0.0511 (10)	-0.0069 (8)	-0.0102 (8)	-0.0071 (8)
C6	0.0348 (8)	0.0415 (8)	0.0452 (10)	-0.0051 (7)	0.0017 (7)	-0.0012 (7)
C7	0.0230 (6)	0.0369 (8)	0.0373 (8)	0.0027 (6)	0.0001 (6)	-0.0021 (6)

*Geometric parameters (Å, °)*

S1—O4	1.4377 (13)	C11—H11B	0.9600
S1—N1	1.6086 (14)	C11—H11C	0.9600

S1—C2	1.7731 (15)	C11—H11A	0.9600
S1—O3	1.4244 (13)	C12—H12A	0.9600
O5—C8	1.211 (3)	C12—H12C	0.9600
O1—C7	1.2416 (19)	C12—H12B	0.9600
O2—C7	1.2551 (17)	C13—H13A	0.9600
N2—C10	1.507 (2)	C13—H13B	0.9600
N2—H2B	0.8900	C13—H13C	0.9600
N2—H2C	0.8900	C1—C7	1.518 (2)
N2—H2A	0.8900	C1—C2	1.405 (2)
N1—H1B	0.85 (2)	C1—C6	1.388 (2)
N1—H1A	0.80 (2)	C2—C3	1.380 (2)
C8—C9	1.503 (3)	C3—C4	1.382 (3)
C8—C13	1.490 (3)	C4—C5	1.374 (3)
C9—C10	1.525 (3)	C5—C6	1.383 (3)
C10—C11	1.514 (3)	C3—H3	0.9300
C10—C12	1.521 (2)	C4—H4	0.9300
C9—H9A	0.9700	C5—H5	0.9300
C9—H9B	0.9700	C6—H6	0.9300
S1…O2	3.1262 (12)	H1A…O4 <sup>iv</sup>	2.21 (2)
O1…N2	2.8124 (18)	H1A…C4 <sup>ii</sup>	3.01 (2)
O1…N2 <sup>i</sup>	2.9878 (17)	H1B…O2	2.10 (2)
O2…N2 <sup>ii</sup>	2.7622 (18)	H1B…C7	2.95 (2)
O2…S1	3.1262 (12)	H2A…O1	1.9400
O2…N1	2.848 (2)	H2A…H12A	2.4100
O2…O3	3.0227 (15)	H2A…C7	2.8600
O3…C7	2.9683 (18)	H2A…H9A	2.4300
O3…O2	3.0227 (15)	H2B…O2 <sup>vii</sup>	1.8700
O4…C4 <sup>iii</sup>	3.235 (2)	H2B…H11B	2.4400
O4…N1 <sup>iv</sup>	3.008 (2)	H2B…H12C	2.4200
O5…C11	3.213 (3)	H2B…C7 <sup>vii</sup>	2.7900
O5…C13 <sup>v</sup>	3.255 (3)	H2C…O1 <sup>v</sup>	2.5000
O5…C8 <sup>v</sup>	3.189 (3)	H2C…H11A	2.4300
O5…N2	2.824 (2)	H2C…O5	2.1800
O1…H9A	2.6900	H2C…C8	2.7100
O1…H2C <sup>i</sup>	2.5000	H3…H3 <sup>iii</sup>	2.5900
O1…H2A	1.9400	H3…O4	2.4700
O1…H6	2.6800	H4…O4 <sup>iii</sup>	2.7000
O1…H11B <sup>ii</sup>	2.9000	H5…C6 <sup>v</sup>	2.9900
O2…H2B <sup>ii</sup>	1.8700	H5…H13A <sup>ix</sup>	2.5500
O2…H1B	2.10 (2)	H6…C7 <sup>v</sup>	2.7800
O2…H12C <sup>ii</sup>	2.9000	H6…O1	2.6800
O2…H6 <sup>i</sup>	2.5200	H6…O2 <sup>v</sup>	2.5200
O3…H12B <sup>vi</sup>	2.6900	H9A…O1	2.6900
O3…H11C <sup>vi</sup>	2.8600	H9A…H2A	2.4300
O3…H9B <sup>vi</sup>	2.7000	H9A…H11B <sup>ii</sup>	2.5900
O3…H12A	2.7600	H9A…H12A	2.4400
O4…H13B <sup>vi</sup>	2.8100	H9B…H13B	2.4800

O4...H3	2.4700	H9B...O3 <sup>x</sup>	2.7000
O4...H4 <sup>iii</sup>	2.7000	H9B...H11C	2.5600
O4...H1A <sup>iv</sup>	2.21 (2)	H9B...H12B	2.4900
O5...H11A	2.6400	H11A...O5	2.6400
O5...H2C	2.1800	H11A...H2C	2.4300
O5...H11A <sup>i</sup>	2.8300	H11A...O5 <sup>v</sup>	2.8300
O5...H13C <sup>v</sup>	2.8400	H11A...C8	2.7600
N1...O2	2.848 (2)	H11B...H9A <sup>vii</sup>	2.5900
N1...C4 <sup>ii</sup>	3.407 (2)	H11B...H12C	2.4500
N1...O4 <sup>iv</sup>	3.008 (2)	H11B...O1 <sup>vii</sup>	2.9000
N2...O1	2.8124 (18)	H11B...H2B	2.4400
N2...O1 <sup>v</sup>	2.9878 (17)	H11C...H9B	2.5600
N2...O2 <sup>vii</sup>	2.7622 (18)	H11C...H12B	2.5600
N2...O5	2.824 (2)	H11C...O3 <sup>x</sup>	2.8600
C4...N1 <sup>vii</sup>	3.407 (2)	H12A...H9A	2.4400
C4...O4 <sup>iii</sup>	3.235 (2)	H12A...H2A	2.4100
C7...O3	2.9683 (18)	H12A...O3	2.7600
C8...O5 <sup>i</sup>	3.189 (3)	H12B...H9B	2.4900
C11...O5	3.213 (3)	H12B...O3 <sup>x</sup>	2.6900
C13...O5 <sup>i</sup>	3.255 (3)	H12B...H11C	2.5600
C4...H13C <sup>viii</sup>	3.0500	H12C...O2 <sup>vii</sup>	2.9000
C4...H1A <sup>vii</sup>	3.01 (2)	H12C...H2B	2.4200
C6...H5 <sup>i</sup>	2.9900	H12C...H11B	2.4500
C7...H1B	2.95 (2)	H13A...H5 <sup>xi</sup>	2.5500
C7...H2B <sup>ii</sup>	2.7900	H13B...H9B	2.4800
C7...H2A	2.8600	H13B...O4 <sup>x</sup>	2.8100
C7...H6 <sup>i</sup>	2.7800	H13C...O5 <sup>i</sup>	2.8400
C8...H2C	2.7100	H13C...C4 <sup>xii</sup>	3.0500
C8...H11A	2.7600		
O3—S1—C2	107.65 (7)	C10—C11—H11B	109.00
O3—S1—O4	118.44 (7)	H12A—C12—H12C	109.00
O3—S1—N1	108.25 (9)	H12A—C12—H12B	109.00
N1—S1—C2	108.12 (8)	C10—C12—H12C	109.00
O4—S1—N1	106.58 (9)	H12B—C12—H12C	109.00
O4—S1—C2	107.44 (7)	C10—C12—H12A	109.00
H2B—N2—H2C	109.00	C10—C12—H12B	109.00
H2A—N2—H2C	109.00	C8—C13—H13A	109.00
H2A—N2—H2B	109.00	H13A—C13—H13B	109.00
C10—N2—H2A	109.00	C8—C13—H13B	109.00
C10—N2—H2B	109.00	C8—C13—H13C	109.00
C10—N2—H2C	109.00	H13B—C13—H13C	109.00
H1A—N1—H1B	121 (2)	H13A—C13—H13C	109.00
S1—N1—H1B	109.0 (13)	C6—C1—C7	117.63 (13)
S1—N1—H1A	109.2 (14)	C2—C1—C7	124.66 (12)
C9—C8—C13	116.4 (2)	C2—C1—C6	117.57 (13)
O5—C8—C9	121.92 (18)	S1—C2—C1	120.76 (10)
O5—C8—C13	121.7 (2)	S1—C2—C3	118.30 (12)



C8—C9—C10	116.54 (16)	C1—C2—C3	120.91 (14)
N2—C10—C9	108.40 (13)	C2—C3—C4	120.16 (16)
C9—C10—C12	110.03 (15)	C3—C4—C5	119.76 (17)
C11—C10—C12	110.46 (15)	C4—C5—C6	120.26 (17)
N2—C10—C11	108.28 (14)	C1—C6—C5	121.28 (16)
N2—C10—C12	107.21 (13)	O1—C7—O2	126.17 (15)
C9—C10—C11	112.29 (15)	O1—C7—C1	117.67 (12)
C8—C9—H9B	108.00	O2—C7—C1	116.02 (13)
C8—C9—H9A	108.00	C2—C3—H3	120.00
H9A—C9—H9B	107.00	C4—C3—H3	120.00
C10—C9—H9A	108.00	C3—C4—H4	120.00
C10—C9—H9B	108.00	C5—C4—H4	120.00
C10—C11—H11C	109.00	C4—C5—H5	120.00
H11B—C11—H11C	109.00	C6—C5—H5	120.00
H11A—C11—H11B	109.00	C1—C6—H6	119.00
H11A—C11—H11C	109.00	C5—C6—H6	119.00
C10—C11—H11A	109.00		
O4—S1—C2—C1	-168.62 (11)	C7—C1—C2—S1	-8.69 (19)
O4—S1—C2—C3	9.43 (15)	C7—C1—C2—C3	173.31 (14)
N1—S1—C2—C1	76.71 (14)	C2—C1—C6—C5	2.4 (2)
N1—S1—C2—C3	-105.24 (14)	C7—C1—C6—C5	-173.44 (16)
O3—S1—C2—C1	-40.02 (13)	C2—C1—C7—O1	135.91 (15)
O3—S1—C2—C3	138.03 (13)	C2—C1—C7—O2	-48.2 (2)
O5—C8—C9—C10	29.1 (3)	C6—C1—C7—O1	-48.5 (2)
C13—C8—C9—C10	-152.4 (2)	C6—C1—C7—O2	127.34 (15)
C8—C9—C10—C12	-179.37 (16)	S1—C2—C3—C4	-177.83 (14)
C8—C9—C10—N2	-62.4 (2)	C1—C2—C3—C4	0.2 (2)
C8—C9—C10—C11	57.2 (2)	C2—C3—C4—C5	1.7 (3)
C6—C1—C2—S1	175.74 (12)	C3—C4—C5—C6	-1.5 (3)
C6—C1—C2—C3	-2.3 (2)	C4—C5—C6—C1	-0.6 (3)

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

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A $\cdots$ O4 <sup>iv</sup>	0.80 (2)	2.21 (2)	3.008 (2)	172 (2)
N1—H1B $\cdots$ O2	0.85 (2)	2.10 (2)	2.848 (2)	145.4 (18)
N2—H2A $\cdots$ O1	0.89	1.94	2.8124 (18)	167
N2—H2B $\cdots$ O2 <sup>vii</sup>	0.89	1.87	2.7622 (18)	176
N2—H2C $\cdots$ O5	0.89	2.18	2.824 (2)	129
N2—H2C $\cdots$ O1 <sup>v</sup>	0.89	2.50	2.9878 (17)	115
C3—H3 $\cdots$ O4	0.93	2.47	2.870 (2)	106
C6—H6 $\cdots$ O2 <sup>v</sup>	0.93	2.52	3.444 (2)	170

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