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1,4-Bis(4,5-dihydro-1H-imidazol-2-yl)-benzene-4-aminobenzenesulfonic acid-water (1/2/2)

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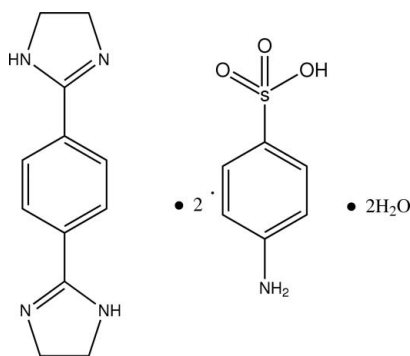
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 Key indicators: single-crystal X-ray study; $T = 273$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.045; wR factor = 0.140; data-to-parameter ratio = 12.9.

The asymmetric unit of the title compound, $\text{C}_{12}\text{H}_{14}\text{N}_4 \cdot 2\text{C}_6\text{H}_7\text{NO}_3\text{S} \cdot 2\text{H}_2\text{O}$, contains one half of a centrosymmetric 1,4-bis(4,5-dihydro-1H-imidazol-2-yl)benzene (bib) molecule, one 4-aminobenzenesulfonic acid molecule and one water molecule. In the bib molecule, the imidazole ring adopts an envelope conformation. The benzene rings of bib and 4-aminobenzenesulfonic acid are oriented at a dihedral angle of $21.89(4)^\circ$. In the crystal structure, intermolecular $\text{N}-\text{H} \cdots \text{O}$, $\text{O}-\text{H} \cdots \text{N}$ and $\text{O}-\text{H} \cdots \text{O}$ interactions link the molecules into a three-dimensional network. Weak $\pi-\pi$ contacts between the benzene and imidazole rings and between the benzene rings [centroid-centroid distances = $3.895(1)$ and $3.833(1)$ Å, respectively] may further stabilize the structure.

Related literature

For general background, see: Jeffrey (1997); Thaimattam *et al.* (1998). For related structures, see: Ren *et al.* (2004a,b, 2007, 2009). For imidazole bond lengths, see: Haga *et al.* (1996); Hammes *et al.* (2005).



Experimental

Crystal data

$\text{C}_{12}\text{H}_{14}\text{N}_4 \cdot 2\text{C}_6\text{H}_7\text{NO}_3\text{S} \cdot 2\text{H}_2\text{O}$	$V = 2698.5(4) \text{ \AA}^3$
$M_r = 596.70$	$Z = 4$
Orthorhombic, $Pbca$	Mo $K\alpha$ radiation
$a = 13.6306(11) \text{ \AA}$	$\mu = 0.26 \text{ mm}^{-1}$
$b = 12.698(1) \text{ \AA}$	$T = 273 \text{ K}$
$c = 15.5907(13) \text{ \AA}$	$0.15 \times 0.12 \times 0.10 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer	11859 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 1998)	2346 independent reflections
$T_{\min} = 0.962$, $T_{\max} = 0.975$	1856 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.036$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$	182 parameters
$wR(F^2) = 0.140$	All H-atom parameters refined
$S = 1.08$	$\Delta\rho_{\text{max}} = 0.45 \text{ e \AA}^{-3}$
2346 reflections	$\Delta\rho_{\text{min}} = -0.42 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{O3}-\text{H3A} \cdots \text{O4}$	0.82	2.00	2.809 (3)	168
$\text{N1}-\text{H1A} \cdots \text{O2}^{\text{i}}$	0.86	2.22	2.960 (3)	145
$\text{N1}-\text{H1B} \cdots \text{O3}^{\text{ii}}$	0.86	2.43	3.200 (3)	150
$\text{N1}-\text{H1B} \cdots \text{O1}^{\text{ii}}$	0.86	2.46	3.170 (4)	141
$\text{N2}-\text{H1} \cdots \text{O3}^{\text{iii}}$	0.86	2.07	2.897 (3)	161
$\text{O4}-\text{H4A} \cdots \text{N3}^{\text{iv}}$	0.85	2.08	2.760 (3)	136
$\text{O4}-\text{H4B} \cdots \text{O1}^{\text{v}}$	0.85	2.20	2.817 (3)	130

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

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL and PLATON (Spek, 2009); software used to prepare material for publication: SHELXTL and PLATON.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2751).

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supporting information

Acta Cryst. (2009). E65, o2221–o2222 [doi:10.1107/S1600536809032504]

1,4-Bis(4,5-dihydro-1*H*-imidazol-2-yl)benzene–4-aminobenzenesulfonic acid–water (1/2/2)

Shao-Ming Shang, Chun-Xia Ren, Xin Wang, Lu-De Lu and Xu-Jie Yang

S1. Comment

Attention has recently focused on the use of supramolecular interactions such as hydrogen bonding and π – π interactions, in addition to coordinate bonds, in the controlled assembly of supramolecular architectures (Jeffrey, 1997). Hydrogen bonds often play a dominant role in crystal engineering because of their combine strength with directionality (Thaimattam *et al.*, 1998). On the other hand, supramolecular systems sustained by soft connections, such as hydrogen bonds, are comparatively more flexible and sensitive to the chemical environment. Consequently hydrogen-bond sustained systems are less designable and remain to be further investigated. We described previously a number of such metal complexes, including imidazole ligand, and have concluded that hydrogen bonding involving this group influences the geometry around the metal atom and the crystallization mechanism (Ren *et al.*, 2004a; Ren *et al.*, 2004b; Ren *et al.*, 2007; Ren *et al.*, 2009). We reported herein the synthesis and crystal structure of the title compound.

The asymmetric unit of the title compound contains one-half of 1,4-bis(4,5-di-hydro-1*H*-imidazol-2-yl)benzene (bib) ligand, one 4-aminobenzenesulfonic acid (SA) and one water molecules. In bib, the imidazole ring B (N2/N3/C7–C9) adopts envelope conformation with atom C8 displaced by $-0.185(3)\text{Å}$ from the plane of the other ring atoms. Rings A (C1–C6) and C (C10/C11/C12/C10'/C11'/C12') [symmetry code ('): $1 - x, 2 - y, 1 - z$] are, of course, planar and they are oriented at a dihedral angle of $21.89(4)^\circ$.

In the crystal structure, intramolecular O–H \cdots O and intermolecular N–H \cdots O, O–H \cdots N and O–H \cdots O interactions (Table 1) link the molecules into a three-dimensional network (Fig. 2), in which they may be effective in the stabilization of the structure. The π – π contacts between the benzene and imidazole rings and between the benzene rings, Cg1—Cg2 and Cg1—Cg3, [where Cg1, Cg2 and Cg3 are centroids of the rings A (C1–C6), B (N2/N3/C7–C9) and C (C10/C11/C12/C10'/C11'/C12'), respectively] may further stabilize the structure, with centroid-centroid distances of $3.895(1)$ and $3.833(1)\text{Å}$, respectively.

S2. Experimental

For the preparation of 1,4-bis(4,5-dihydro-1*H*-imidazol-2-yl)benzene, (bib), 1,4-benzenedicarboxylic acid (2.31 g, 13.9 mmol), ethylenediamine (3.70 ml, 50 mmol), ethylenediamine dihydrochloride(6.64 g, 50 mmol) and toluene-*p*-sulfonic acid (0.208 g, 1.09 mmol) were added to the solvent of ethyleneglycol (20 ml), and the mixture was refluxed for 3 h. About half of the ethylene glycol solvent was then slowly removed by distillation. The residue was dissolved in a mixture of water (40 ml) and concentrated HCl (11 *M*, 3 ml). The addition of 50% aqueous NaOH gave a yellow precipitate that was purified by recrystallization. The ligand bib was obtained in 83% based on 1,4-benzenedicarboxylic acid (*ca* 2.50 g). Found: C 66.98; H 6.92; N 26.08%. Calc. for C₁₂H₁₄N₄: C 67.27; H 6.59; N 26.15%. Main IR bonds (KBr, cm⁻¹): 3188*m*, 2936*m*, 2866*m*, 1606 *s*, 1532 *s*, 1466 *s*, 1345*m*, 1270 *s*, 1191*w*, 1080*w*, 981*m*, 907*w*, 767*w*, 687*m*. For the preparation of the title compound, to a solution of bib (0.043 g, 0.2 mmol) in MeOH (15 ml), an aqueous solution (5 ml)

of SA (0.068 g, 0.4 mmol) was added. The solution was allowed at room temperature in air for 3 d by slow evaporation. Large yellow prismatic crystals were obtained, which were collected by filtration, washed with water and dried in vacuum desiccator over silica gel (0.047 g, 54%). Main IR bonds (KBr, cm^{-1}): 3424 m , 3354 m , 3249 w , 1655 w , 1603 m , 1507 m , 1119 m , 1024 s , 1001 m , 698 m , 569 w .

S3. Refinement

H atoms were positioned geometrically, with N-H = 0.86 Å (for NH and NH₂), O-H = 0.82 Å (for OH) and 0.85 Å (for H₂O) and C-H = 0.93 and 0.97 Å for aromatic and methylene H, respectively, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C,N,O})$, where $x = 1.5$ for OH H and $x = 1.2$ for all other H atoms.

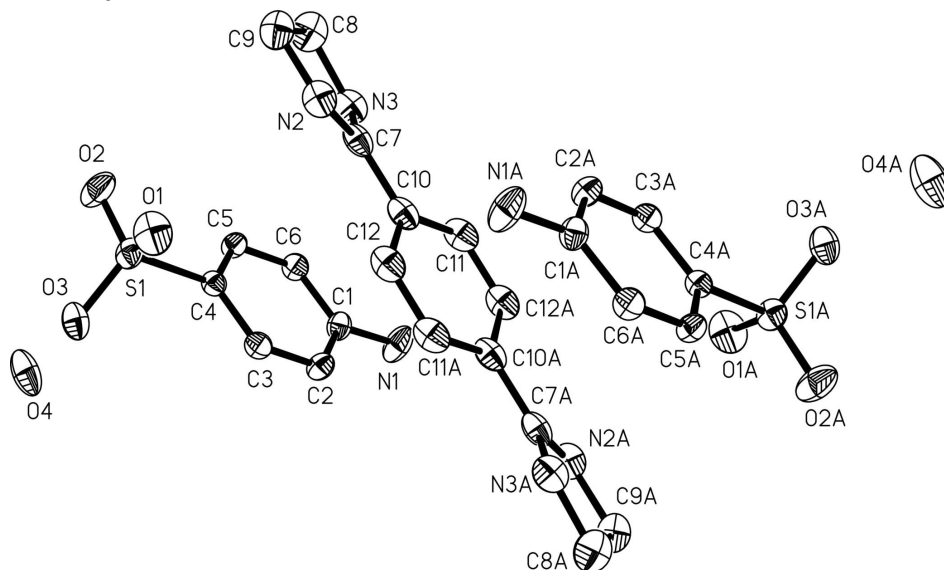
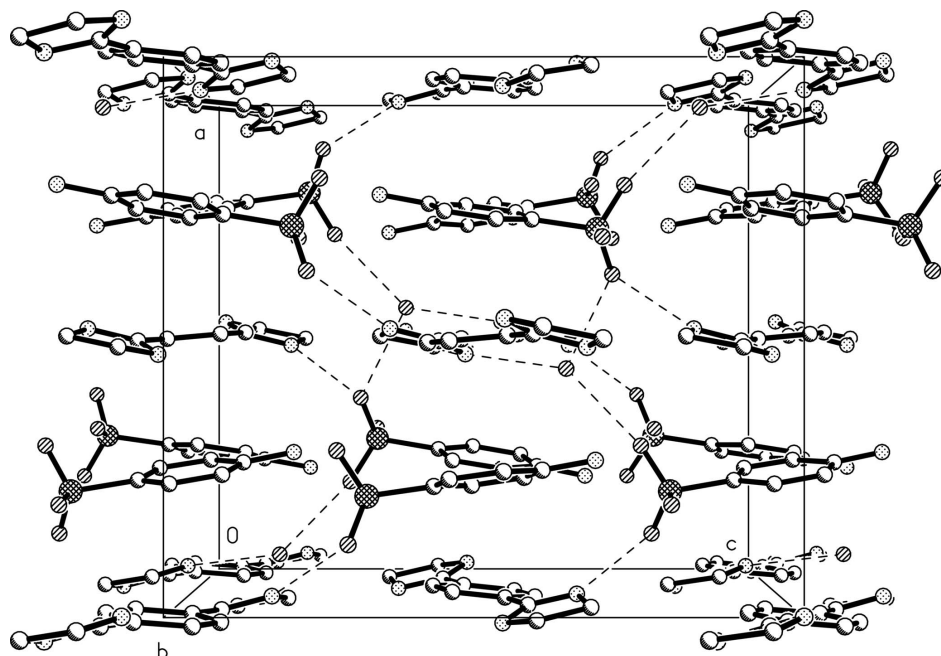


Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Hydrogen bond is shown as dashed line [symmetry code ('): 1 - x, 2 - y, 1 - z].

**Figure 2**

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

1,4-Bis(4,5-dihydro-1H-imidazol-2-yl)benzene-4-aminobenzenesulfonic acid-water (1/2/2)

Crystal data

$C_{12}H_{14}N_4 \cdot 2C_6H_7NO_3S \cdot 2H_2O$

$M_r = 596.70$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 13.6306$ (11) Å

$b = 12.698$ (1) Å

$c = 15.5907$ (13) Å

$V = 2698.5$ (4) Å³

$Z = 4$

$F(000) = 1256$

$D_x = 1.469$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3721 reflections

$\theta = 2.6$ – 26.3°

$\mu = 0.26$ mm⁻¹

$T = 273$ K

Block, yellow

$0.15 \times 0.12 \times 0.10$ mm

Data collection

Bruker SMART CCD area-detector

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 1998)

$T_{\min} = 0.962$, $T_{\max} = 0.975$

11859 measured reflections

2346 independent reflections

1856 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.036$

$\theta_{\max} = 25.1^\circ$, $\theta_{\min} = 2.6^\circ$

$h = -16 \rightarrow 15$

$k = -15 \rightarrow 15$

$l = -18 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.045$

$wR(F^2) = 0.140$

$S = 1.08$

2346 reflections

182 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

All H-atom parameters refined

$$w = 1/[\sigma^2(F_o^2) + (0.0724P)^2 + 1.4974P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.45 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.42 \text{ e } \text{\AA}^{-3}$$

Extinction correction: *SHELXTL* (Sheldrick, 2008), $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0010 (5)

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.20619 (5)	0.82812 (5)	0.31074 (4)	0.0423 (3)
O1	0.29197 (15)	0.8625 (2)	0.26382 (13)	0.0688 (7)
O2	0.1875 (2)	0.71668 (16)	0.30647 (13)	0.0681 (7)
O3	0.12151 (14)	0.88982 (15)	0.28322 (11)	0.0536 (5)
H3A	0.0729	0.8714	0.3104	0.080*
O4	-0.06007 (17)	0.8498 (2)	0.36193 (13)	0.0774 (8)
H4A	-0.0646	0.8028	0.4006	0.093*
H4B	-0.1105	0.8846	0.3470	0.093*
N1	0.2770 (2)	0.9242 (2)	0.67696 (14)	0.0615 (8)
H1A	0.2775	0.8740	0.7140	0.074*
H1B	0.2884	0.9878	0.6930	0.074*
N2	0.51620 (17)	0.7721 (2)	0.34661 (15)	0.0518 (6)
H1	0.5368	0.8180	0.3103	0.062*
N3	0.46812 (18)	0.70826 (19)	0.46926 (16)	0.0530 (6)
C1	0.25816 (19)	0.90302 (19)	0.59303 (15)	0.0384 (6)
C2	0.2594 (2)	0.98296 (19)	0.53090 (16)	0.0398 (6)
H2	0.2705	1.0522	0.5476	0.048*
C3	0.24456 (19)	0.96036 (18)	0.44548 (15)	0.0368 (6)
H3	0.2470	1.0141	0.4050	0.044*
C4	0.22603 (17)	0.85807 (18)	0.41959 (15)	0.0327 (5)
C5	0.22330 (18)	0.77832 (18)	0.48013 (15)	0.0362 (6)
H5	0.2105	0.7095	0.4631	0.043*
C6	0.2394 (2)	0.80063 (19)	0.56551 (15)	0.0392 (6)
H6	0.2378	0.7463	0.6055	0.047*
C7	0.49343 (18)	0.7937 (2)	0.42682 (17)	0.0441 (7)
C8	0.4824 (3)	0.6141 (3)	0.4154 (2)	0.0657 (9)
H8A	0.4241	0.5703	0.4149	0.079*
H8B	0.5377	0.5726	0.4351	0.079*
C9	0.5021 (3)	0.6610 (3)	0.3270 (2)	0.0604 (8)

H9A	0.5604	0.6307	0.3013	0.073*
H9B	0.4468	0.6506	0.2889	0.073*
C10	0.49700 (19)	0.8996 (2)	0.46426 (16)	0.0446 (7)
C11	0.5064 (2)	0.9132 (2)	0.55335 (17)	0.0513 (7)
H11	0.5105	0.8546	0.5890	0.062*
C12	0.4905 (2)	0.9878 (2)	0.41216 (17)	0.0514 (7)
H12	0.4839	0.9796	0.3532	0.062*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0514 (5)	0.0491 (4)	0.0264 (4)	-0.0001 (3)	0.0007 (3)	-0.0023 (3)
O1	0.0604 (14)	0.1066 (17)	0.0396 (12)	-0.0006 (13)	0.0178 (10)	0.0025 (11)
O2	0.114 (2)	0.0497 (12)	0.0407 (12)	-0.0013 (12)	-0.0076 (11)	-0.0109 (9)
O3	0.0497 (12)	0.0712 (13)	0.0399 (10)	-0.0024 (10)	-0.0102 (9)	0.0053 (9)
O4	0.0590 (14)	0.128 (2)	0.0449 (13)	0.0017 (14)	-0.0103 (10)	0.0270 (13)
N1	0.097 (2)	0.0549 (15)	0.0321 (12)	-0.0210 (14)	-0.0048 (12)	-0.0035 (10)
N2	0.0504 (14)	0.0678 (16)	0.0371 (13)	-0.0018 (12)	0.0070 (11)	0.0087 (11)
N3	0.0438 (14)	0.0646 (15)	0.0507 (14)	-0.0019 (12)	0.0108 (11)	0.0103 (12)
C1	0.0396 (14)	0.0445 (13)	0.0312 (13)	-0.0052 (12)	0.0007 (11)	-0.0021 (10)
C2	0.0468 (16)	0.0335 (12)	0.0390 (14)	-0.0056 (11)	0.0020 (11)	-0.0019 (10)
C3	0.0389 (14)	0.0360 (12)	0.0356 (13)	0.0004 (11)	0.0006 (11)	0.0053 (10)
C4	0.0317 (13)	0.0359 (12)	0.0304 (12)	0.0021 (10)	0.0005 (10)	0.0013 (9)
C5	0.0457 (15)	0.0315 (12)	0.0314 (13)	-0.0014 (11)	-0.0010 (10)	-0.0005 (9)
C6	0.0466 (15)	0.0374 (12)	0.0335 (13)	-0.0011 (12)	-0.0016 (12)	0.0068 (10)
C7	0.0290 (14)	0.0668 (17)	0.0366 (14)	0.0049 (13)	0.0043 (11)	0.0116 (12)
C8	0.063 (2)	0.071 (2)	0.063 (2)	-0.0182 (17)	0.0129 (16)	0.0054 (16)
C9	0.058 (2)	0.072 (2)	0.0507 (18)	-0.0093 (16)	0.0045 (15)	-0.0012 (15)
C10	0.0330 (14)	0.0646 (17)	0.0361 (14)	0.0076 (13)	0.0063 (11)	0.0110 (12)
C11	0.0509 (17)	0.0654 (18)	0.0377 (15)	0.0104 (14)	0.0065 (12)	0.0146 (13)
C12	0.0494 (17)	0.0729 (19)	0.0318 (14)	0.0101 (15)	0.0042 (12)	0.0094 (13)

Geometric parameters (Å, °)

S1—O2	1.439 (2)	C3—C4	1.383 (3)
S1—O1	1.447 (2)	C3—H3	0.9300
S1—O3	1.460 (2)	C4—C5	1.385 (3)
S1—C4	1.760 (2)	C5—C6	1.379 (3)
O3—H3A	0.8200	C5—H5	0.9300
O4—H4A	0.8501	C6—H6	0.9300
O4—H4B	0.8501	C7—C10	1.467 (4)
N1—C1	1.360 (3)	C8—C9	1.525 (4)
N1—H1A	0.8600	C8—H8A	0.9700
N1—H1B	0.8600	C8—H8B	0.9700
N2—C7	1.317 (3)	C9—H9A	0.9700
N2—C9	1.455 (4)	C9—H9B	0.9700
N2—H1	0.8600	C10—C12	1.387 (4)
N3—C7	1.317 (3)	C10—C11	1.405 (4)

N3—C8	1.474 (4)	C11—C12 ⁱ	1.368 (4)
C1—C6	1.393 (3)	C11—H11	0.9300
C1—C2	1.403 (3)	C12—C11 ⁱ	1.368 (4)
C2—C3	1.377 (3)	C12—H12	0.9300
C2—H2	0.9300		
O2—S1—O1	114.61 (15)	C4—C5—H5	119.9
O2—S1—O3	111.99 (14)	C5—C6—C1	121.2 (2)
O1—S1—O3	109.17 (13)	C5—C6—H6	119.4
O2—S1—C4	106.51 (12)	C1—C6—H6	119.4
O1—S1—C4	107.34 (13)	N3—C7—N2	111.6 (3)
O3—S1—C4	106.79 (11)	N3—C7—C10	124.3 (2)
S1—O3—H3A	109.5	N2—C7—C10	124.1 (2)
H4A—O4—H4B	120.0	N3—C8—C9	102.8 (3)
C1—N1—H1A	120.0	N3—C8—H8A	111.2
C1—N1—H1B	120.0	C9—C8—H8A	111.2
H1A—N1—H1B	120.0	N3—C8—H8B	111.2
C7—N2—C9	111.7 (2)	C9—C8—H8B	111.2
C7—N2—H1	124.2	H8A—C8—H8B	109.1
C9—N2—H1	124.2	N2—C9—C8	102.3 (2)
C7—N3—C8	110.3 (2)	N2—C9—H9A	111.3
N1—C1—C6	121.0 (2)	C8—C9—H9A	111.3
N1—C1—C2	121.2 (2)	N2—C9—H9B	111.3
C6—C1—C2	117.7 (2)	C8—C9—H9B	111.3
C3—C2—C1	121.0 (2)	H9A—C9—H9B	109.2
C3—C2—H2	119.5	C12—C10—C11	119.0 (3)
C1—C2—H2	119.5	C12—C10—C7	120.4 (2)
C2—C3—C4	120.3 (2)	C11—C10—C7	120.6 (2)
C2—C3—H3	119.8	C12 ⁱ —C11—C10	120.3 (3)
C4—C3—H3	119.8	C12 ⁱ —C11—H11	119.9
C3—C4—C5	119.5 (2)	C10—C11—H11	119.9
C3—C4—S1	120.81 (18)	C11 ⁱ —C12—C10	120.7 (3)
C5—C4—S1	119.67 (18)	C11 ⁱ —C12—H12	119.7
C6—C5—C4	120.2 (2)	C10—C12—H12	119.7
C6—C5—H5	119.9		

Symmetry code: (i) $-x+1, -y+2, -z+1$.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3A \cdots O4	0.82	2.00	2.809 (3)	168
N1—H1A \cdots O2 ⁱⁱ	0.86	2.22	2.960 (3)	145
N1—H1B \cdots O3 ⁱⁱⁱ	0.86	2.43	3.200 (3)	150
N1—H1B \cdots O1 ⁱⁱⁱ	0.86	2.46	3.170 (4)	141
N2—H1 \cdots O3 ^{iv}	0.86	2.07	2.897 (3)	161

O4—H4A···N3 ^v	0.85	2.08	2.760 (3)	136
O4—H4B···O1 ^{vi}	0.85	2.20	2.817 (3)	130

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