

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

4-Amino-*N*-(4,6-dimethylpyrimidin-2-yl)-benzenesulfonamide–1,4-diazabicyclo[2.2.2]octane (2/1)

 Hadi D. Arman,^a Trupta Kaulgud^a and Edward R. T. Tiekink^{b*}

^aDepartment of Chemistry, The University of Texas at San Antonio, One UTSA Circle, San Antonio, Texas 78249-0698, USA, and ^bDepartment of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: edward.tiekink@gmail.com

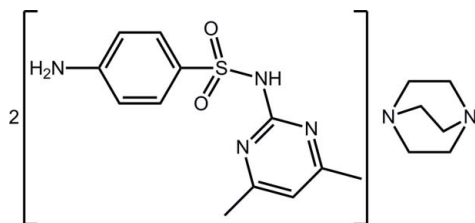
Received 30 September 2013; accepted 1 October 2013

Key indicators: single-crystal X-ray study; $T = 98$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.044; wR factor = 0.115; data-to-parameter ratio = 16.5.

The asymmetric unit of the title co-crystal, $\text{C}_{12}\text{H}_{14}\text{N}_4\text{O}_2\text{S} \cdot 0.5\text{C}_6\text{H}_{12}\text{N}_2$, comprises the sulfonamide molecule and half a molecule of 1,4-diazabicyclo[2.2.2]octane (DABCO), the latter being disposed about a crystallographic twofold rotation axis. In the sulfonamide molecule, the aromatic rings are almost perpendicular to one another [dihedral angle = $75.01(8)^\circ$]. In the crystal, molecules are connected into a three-molecule aggregate *via* amide–DABCO $\text{N}-\text{H} \cdots \text{N}$ hydrogen bonds, and these are connected into a three-dimensional architecture *via* amino–DABCO $\text{N}-\text{H} \cdots \text{O}$ and amino–pyrimidine $\text{N}-\text{H} \cdots \text{N}$ hydrogen bonds.

Related literature

For the structure of the sulfonamide, see: Tiwari *et al.* (1984). For related studies of co-crystal formation, see: Ellis *et al.* (2009); Arman & Tiekink (2013). For co-crystals of the sulfonamide with carboxylic acids, see: Arman *et al.* (2010); Ghosh *et al.* (2011); Smith & Wermuth (2013).



Experimental

Crystal data

$\text{C}_{12}\text{H}_{14}\text{N}_4\text{O}_2\text{S} \cdot 0.5\text{C}_6\text{H}_{12}\text{N}_2$
 $M_r = 334.42$

Orthorhombic, $Pbcn$
 $a = 26.488(3)$ Å

$b = 9.7886(11)$ Å
 $c = 12.2163(13)$ Å
 $V = 3167.4(6)$ Å³
 $Z = 8$

Mo $K\alpha$ radiation
 $\mu = 0.22$ mm⁻¹
 $T = 98$ K
 $0.35 \times 0.31 \times 0.21$ mm

Data collection

Rigaku AFC12/SATURN724
diffractometer
7965 measured reflections

3616 independent reflections
3264 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.115$
 $S = 0.99$
3616 reflections
219 parameters
3 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.41$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.48$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{N1}-\text{H1N} \cdots \text{N5}^i$	0.88 (2)	1.90 (2)	2.768 (2)	169 (2)
$\text{N4}-\text{H2N} \cdots \text{O2}^{ii}$	0.88 (1)	2.48 (2)	3.058 (2)	124 (2)
$\text{N4}-\text{H2N} \cdots \text{N2}^{ii}$	0.88 (1)	2.59 (2)	3.376 (2)	149 (2)
$\text{N4}-\text{H3N} \cdots \text{O1}^{iii}$	0.88 (2)	2.15 (2)	3.032 (2)	178 (2)

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

Data collection: *CrystalClear* (Molecular Structure Corporation & Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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, 2012) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We are grateful to the Ministry of Higher Education (Malaysia) and the University of Malaya (UM) for funding structural studies through the High-Impact Research scheme (UM-C/HIR-MOHE/SC/03).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2652).

References

- Arman, H. D., Kaulgud, T. & Tiekink, E. R. T. (2010). *Acta Cryst.* **E66**, o2430.
Arman, H. D. & Tiekink, E. R. T. (2013). *Z. Kristallogr. Cryst. Mat.* **228**, 289–294.
Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Ellis, C. A., Miller, M. A., Spencer, J., Zukerman-Schpector, J. & Tiekink, E. R. T. (2009). *CrystEngComm*, **11**, 1352–1361.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Ghosh, S., Bag, P. P. & Reddy, C. M. (2011). *Cryst. Growth Des.* **11**, 3489–3503.
Molecular Structure Corporation & Rigaku (2005). *CrystalClear*. MSC, The Woodlands, Texas, USA, and Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Smith, G. & Wermuth, U. D. (2013). *Acta Cryst.* **E69**, o234.
Tiwari, R. K., Haridas, M. & Singh, T. P. (1984). *Acta Cryst.* **C40**, 655–657.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2013). E69, o1615 [doi:10.1107/S1600536813027037]

4-Amino-*N*-(4,6-dimethylpyrimidin-2-yl)benzenesulfonamide–1,4-diazabicyclo[2.2.2]octane (2/1)

Hadi D. Arman, Trupta Kaulgud and Edward R. T. Tiekink

S1. Comment

The title co-crystal was formed in continuation of on-going structural studies of co-crystals (Ellis *et al.*, 2009; Arman & Tiekink, 2013). While co-crystals of the title sulfonamide with carboxylic acids are known (Arman, *et al.* 2010; Ghosh *et al.* 2011; Smith & Wermuth, 2013), the present investigation appears to be the first describing a co-crystal of the sulfonamide with an amine.

The asymmetric unit of the title compound contains a molecule of the sulfonamide in a general position, and half a molecule of 1,4-diazabicyclo[2.2.2]octane (DABCO) which is disposed about a crystallographic two-fold rotation axis, Fig. 1. The overall shape of the sulfonamide approximates the letter *L* with the dihedral angle between the two aromatic rings being 75.01 (8)°, which compares to 78.1 (6)° found in the parent sulfonamide compound (Tiwari *et al.*, 1984). However, this is a little misleading as there is a difference in the conformation of the two sulfonamides. In the title sulfonamide the SO₂C₆H₄NH₂ residue lies to one side of the pyrimidinyl ring with the remaining O atom, O2, being coplanar giving the *L*-shape, whereas in the parent sulfonamide (Tiwari *et al.*, 1984) the SO₂ O atoms lie to one side of the pyrimidinyl ring and the C₆H₄NH₂ residue to the other.

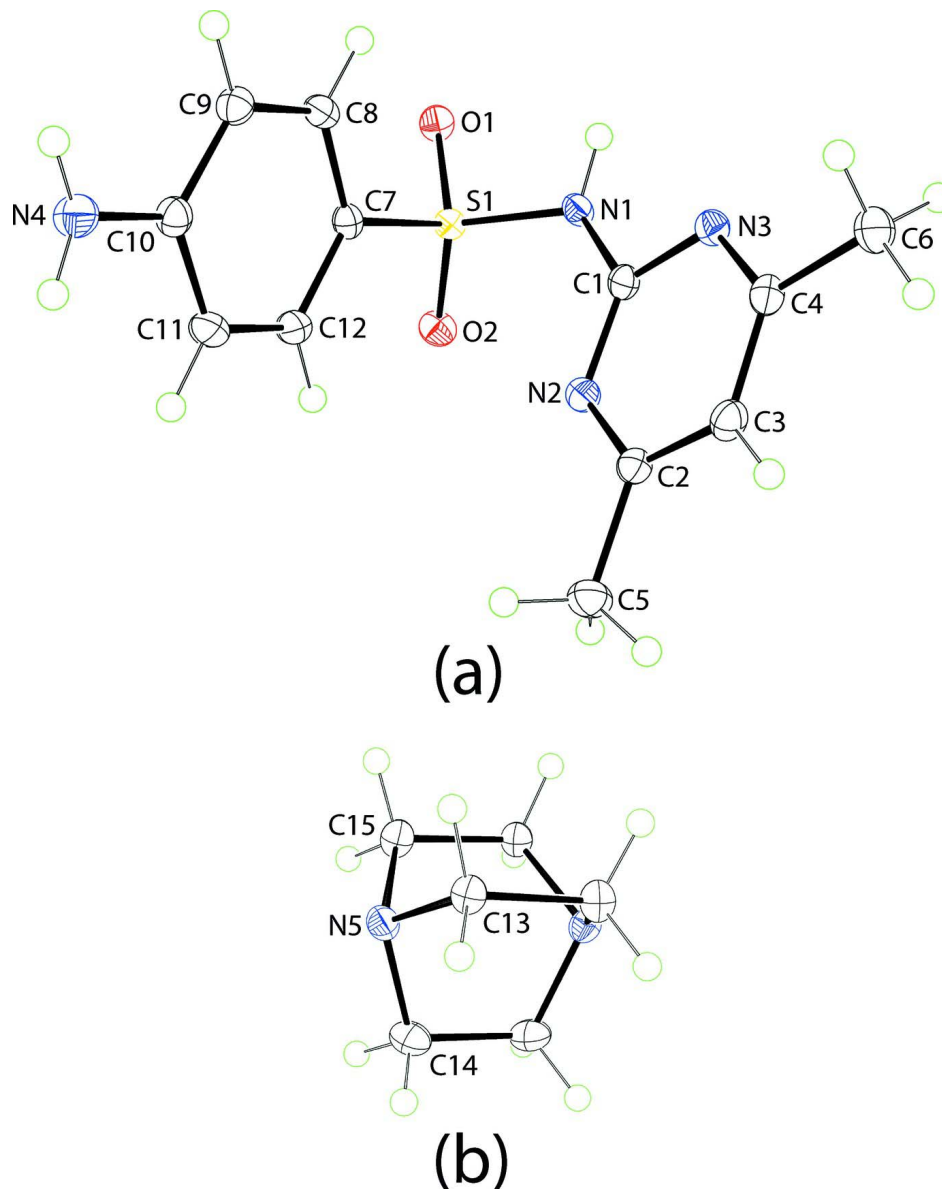
A three-dimensional architecture is formed in the crystal structure by N—H···O and N-H···N hydrogen bonds (Fig. 2 and Table 1). The amide group, N1—H1N, forms a hydrogen bond to a DABCO N atom, N5, so that a three-molecule aggregate results. The amino group H atom, N4—H2N, is bifurcated, forming hydrogen bonds to the sulfonamide atom O2 and to the pyrimidinyl atom N2. The second amino group H atom, N4—H3N, forms a hydrogen bond to the second sulfonamide O atom, O1.

S2. Experimental

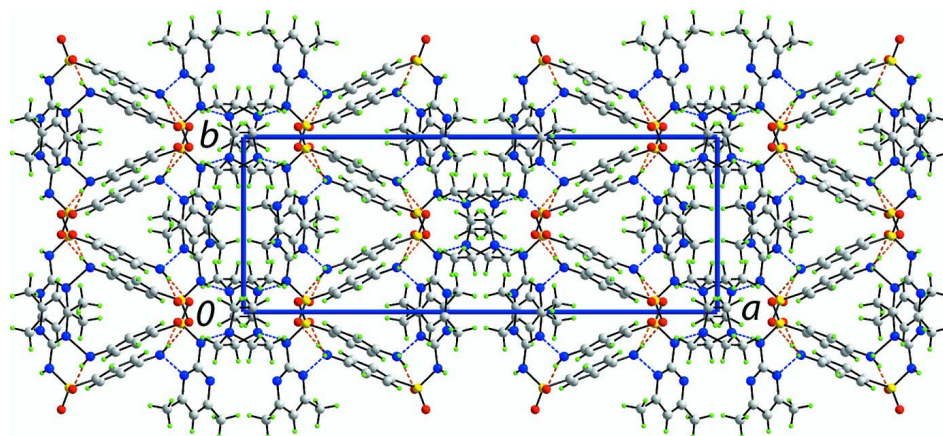
Crystals of the title compound were obtained by the co-crystallization of the sulfonamide (ACROS, 0.18 mmol) and 1,4-diazabicyclo[2.2.2]octane (DABCO; Sigma-Aldrich, 0.10 mmol) in methanol. Block-like colourless crystals were obtained by slow evaporation (M.p. = 479–485 K).

S3. Refinement

The N-bound H-atoms were located in a difference Fourier map and refined with a distance restraint: N—H = 0.88 (1) Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$. The C-bound H-atoms were placed in calculated positions and included in the refinement in the riding model approximation: C—H = 0.95–0.99 Å with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C-methyl})$ and = 1.2 $U_{\text{eq}}(\text{C})$ for other H atoms.

**Figure 1**

Molecular structures of the components of the title compound, with atom labelling: (a) the sulfonamide and (b) DABCO. Displacement ellipsoids are drawn at the 50% probability level.

**Figure 2**

A viewed in projection along the c axis of the crystal packing of the title compound. The N-H \cdots O and N-H \cdots N hydrogen bonds are shown as orange and blue dashed lines, respectively.

4-Amino-*N*-(4,6-dimethylpyrimidin-2-yl)benzenesulfonamide-1,4-diazabicyclo[2.2.2]octane (2/1)

Crystal data

$C_{12}H_{14}N_4O_2S \cdot 0.5C_6H_{12}N_2$

$M_r = 334.42$

Orthorhombic, $Pbcn$

Hall symbol: $-P\ 2n\ 2ab$

$a = 26.488\ (3)\ \text{\AA}$

$b = 9.7886\ (11)\ \text{\AA}$

$c = 12.2163\ (13)\ \text{\AA}$

$V = 3167.4\ (6)\ \text{\AA}^3$

$Z = 8$

$F(000) = 1416$

$D_x = 1.403\ \text{Mg m}^{-3}$

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

Cell parameters from 11130 reflections

$\theta = 2.1\text{--}40.3^\circ$

$\mu = 0.22\ \text{mm}^{-1}$

$T = 98\ \text{K}$

Block, colourless

$0.35 \times 0.31 \times 0.21\ \text{mm}$

Data collection

Rigaku AFC12K/SATURN724
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

7965 measured reflections

3616 independent reflections

3264 reflections with $I > 2\sigma(I)$

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

$\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 2.2^\circ$

$h = -10 \rightarrow 34$

$k = -8 \rightarrow 12$

$l = -15 \rightarrow 10$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.044$

$wR(F^2) = 0.115$

$S = 0.99$

3616 reflections

219 parameters

3 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

$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 2.7774P]$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\text{max}} = 0.41\ \text{e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.48\ \text{e \AA}^{-3}$

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.132415 (15)	0.06565 (4)	0.45540 (3)	0.01333 (12)
O1	0.11533 (5)	-0.05810 (12)	0.50942 (10)	0.0173 (3)
O2	0.13901 (5)	0.05841 (13)	0.33849 (10)	0.0183 (3)
N1	0.09018 (5)	0.17725 (15)	0.48968 (11)	0.0148 (3)
H1N	0.0745 (7)	0.152 (2)	0.5494 (11)	0.018*
N2	0.12538 (5)	0.36203 (15)	0.39075 (11)	0.0160 (3)
N3	0.07074 (6)	0.39389 (15)	0.54545 (11)	0.0160 (3)
N4	0.32337 (6)	0.25427 (17)	0.65547 (13)	0.0218 (3)
H2N	0.3284 (9)	0.250 (2)	0.7267 (8)	0.026*
H3N	0.3418 (8)	0.308 (2)	0.6139 (16)	0.026*
C1	0.09647 (6)	0.31690 (17)	0.47297 (13)	0.0148 (3)
C2	0.12939 (6)	0.49905 (19)	0.38156 (14)	0.0166 (3)
C3	0.10504 (7)	0.58644 (18)	0.45383 (14)	0.0181 (4)
H3	0.1085	0.6827	0.4474	0.022*
C4	0.07545 (7)	0.52934 (18)	0.53588 (14)	0.0169 (3)
C5	0.16111 (8)	0.55243 (19)	0.28925 (15)	0.0226 (4)
H5A	0.1968	0.5353	0.3050	0.034*
H5B	0.1555	0.6509	0.2812	0.034*
H5C	0.1517	0.5060	0.2212	0.034*
C6	0.04783 (7)	0.61545 (19)	0.61820 (15)	0.0223 (4)
H6A	0.0120	0.5909	0.6180	0.033*
H6B	0.0515	0.7122	0.5990	0.033*
H6C	0.0620	0.5995	0.6912	0.033*
C7	0.18946 (6)	0.11773 (17)	0.51417 (13)	0.0144 (3)
C8	0.19655 (6)	0.10442 (17)	0.62712 (14)	0.0159 (3)
H8	0.1710	0.0640	0.6712	0.019*
C9	0.24078 (6)	0.15020 (18)	0.67437 (14)	0.0173 (3)
H9	0.2456	0.1396	0.7509	0.021*
C10	0.27886 (7)	0.21239 (18)	0.61103 (14)	0.0172 (3)
C11	0.27024 (7)	0.2272 (2)	0.49774 (15)	0.0211 (4)
H11	0.2950	0.2707	0.4535	0.025*
C12	0.22649 (7)	0.17962 (19)	0.45039 (14)	0.0192 (4)
H12	0.2216	0.1890	0.3737	0.023*
N5	0.02978 (6)	0.88272 (15)	0.16824 (11)	0.0165 (3)
C13	0.05365 (6)	0.80970 (18)	0.26139 (13)	0.0168 (3)

H13A	0.0862	0.8535	0.2799	0.020*
H13B	0.0604	0.7137	0.2407	0.020*
C14	0.01889 (8)	1.02566 (18)	0.20149 (15)	0.0234 (4)
H14A	0.0043	1.0765	0.1389	0.028*
H14B	0.0506	1.0716	0.2236	0.028*
C15	-0.01819 (6)	0.81388 (19)	0.13850 (14)	0.0180 (3)
H15A	-0.0111	0.7197	0.1133	0.022*
H15B	-0.0347	0.8639	0.0779	0.022*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0140 (2)	0.0121 (2)	0.0139 (2)	-0.00069 (14)	0.00026 (15)	-0.00111 (14)
O1	0.0176 (6)	0.0125 (6)	0.0219 (6)	-0.0016 (5)	0.0005 (5)	0.0000 (5)
O2	0.0206 (6)	0.0201 (6)	0.0142 (6)	-0.0009 (5)	0.0005 (5)	-0.0043 (5)
N1	0.0135 (6)	0.0136 (7)	0.0174 (6)	-0.0002 (5)	0.0036 (5)	0.0003 (5)
N2	0.0162 (7)	0.0152 (7)	0.0166 (7)	0.0004 (5)	0.0013 (6)	0.0005 (6)
N3	0.0162 (7)	0.0162 (7)	0.0157 (6)	0.0013 (6)	0.0006 (5)	-0.0005 (5)
N4	0.0188 (7)	0.0266 (8)	0.0198 (7)	-0.0053 (6)	-0.0043 (6)	0.0018 (6)
C1	0.0123 (7)	0.0162 (8)	0.0158 (7)	0.0002 (6)	-0.0018 (6)	0.0000 (6)
C2	0.0150 (7)	0.0174 (8)	0.0174 (8)	0.0003 (6)	-0.0006 (6)	0.0022 (7)
C3	0.0188 (8)	0.0132 (7)	0.0225 (9)	0.0012 (6)	-0.0007 (7)	0.0011 (6)
C4	0.0158 (8)	0.0172 (8)	0.0176 (8)	0.0027 (6)	-0.0017 (6)	-0.0012 (6)
C5	0.0255 (9)	0.0181 (8)	0.0243 (9)	-0.0014 (7)	0.0057 (8)	0.0028 (7)
C6	0.0246 (9)	0.0188 (8)	0.0234 (9)	0.0047 (7)	0.0034 (7)	-0.0024 (7)
C7	0.0121 (7)	0.0140 (7)	0.0171 (7)	-0.0002 (6)	-0.0002 (6)	-0.0008 (6)
C8	0.0162 (8)	0.0155 (8)	0.0160 (7)	-0.0004 (6)	0.0034 (6)	0.0001 (6)
C9	0.0181 (8)	0.0181 (8)	0.0157 (7)	0.0007 (7)	-0.0015 (6)	-0.0012 (6)
C10	0.0154 (8)	0.0156 (8)	0.0204 (8)	-0.0002 (6)	-0.0008 (7)	0.0006 (7)
C11	0.0163 (8)	0.0255 (9)	0.0214 (8)	-0.0039 (7)	0.0018 (7)	0.0064 (7)
C12	0.0154 (8)	0.0251 (9)	0.0172 (8)	-0.0008 (7)	-0.0005 (7)	0.0053 (7)
N5	0.0166 (7)	0.0174 (7)	0.0154 (6)	-0.0021 (6)	0.0007 (5)	0.0016 (6)
C13	0.0151 (8)	0.0194 (8)	0.0159 (7)	0.0004 (6)	-0.0001 (6)	-0.0003 (6)
C14	0.0300 (10)	0.0144 (8)	0.0258 (9)	-0.0003 (7)	0.0000 (8)	0.0028 (7)
C15	0.0141 (7)	0.0234 (9)	0.0165 (7)	-0.0010 (7)	-0.0009 (6)	-0.0013 (7)

Geometric parameters (Å, °)

S1—O2	1.4406 (12)	C6—H6C	0.9800
S1—O1	1.4518 (12)	C7—C12	1.392 (2)
S1—N1	1.6188 (14)	C7—C8	1.399 (2)
S1—C7	1.7488 (17)	C8—C9	1.381 (2)
N1—C1	1.392 (2)	C8—H8	0.9500
N1—H1N	0.876 (9)	C9—C10	1.409 (2)
N2—C1	1.338 (2)	C9—H9	0.9500
N2—C2	1.350 (2)	C10—C11	1.410 (2)
N3—C4	1.337 (2)	C11—C12	1.377 (2)
N3—C1	1.348 (2)	C11—H11	0.9500

N4—C10	1.361 (2)	C12—H12	0.9500
N4—H2N	0.881 (9)	N5—C15	1.483 (2)
N4—H3N	0.877 (10)	N5—C14	1.485 (2)
C2—C3	1.388 (2)	N5—C13	1.485 (2)
C2—C5	1.500 (2)	C13—C15 ⁱ	1.542 (2)
C3—C4	1.390 (2)	C13—H13A	0.9900
C3—H3	0.9500	C13—H13B	0.9900
C4—C6	1.502 (2)	C14—C14 ⁱ	1.551 (4)
C5—H5A	0.9800	C14—H14A	0.9900
C5—H5B	0.9800	C14—H14B	0.9900
C5—H5C	0.9800	C15—C13 ⁱ	1.542 (2)
C6—H6A	0.9800	C15—H15A	0.9900
C6—H6B	0.9800	C15—H15B	0.9900
O2—S1—O1	116.57 (7)	C12—C7—S1	120.40 (13)
O2—S1—N1	111.93 (8)	C8—C7—S1	119.60 (13)
O1—S1—N1	103.30 (7)	C9—C8—C7	119.74 (16)
O2—S1—C7	108.46 (8)	C9—C8—H8	120.1
O1—S1—C7	109.02 (8)	C7—C8—H8	120.1
N1—S1—C7	107.11 (8)	C8—C9—C10	121.19 (15)
C1—N1—S1	122.82 (12)	C8—C9—H9	119.4
C1—N1—H1N	117.0 (14)	C10—C9—H9	119.4
S1—N1—H1N	110.8 (14)	N4—C10—C11	120.04 (16)
C1—N2—C2	115.81 (15)	N4—C10—C9	122.07 (16)
C4—N3—C1	116.74 (15)	C11—C10—C9	117.86 (16)
C10—N4—H2N	120.7 (15)	C12—C11—C10	120.92 (16)
C10—N4—H3N	115.6 (15)	C12—C11—H11	119.5
H2N—N4—H3N	121 (2)	C10—C11—H11	119.5
N2—C1—N3	126.72 (16)	C11—C12—C7	120.37 (16)
N2—C1—N1	120.18 (15)	C11—C12—H12	119.8
N3—C1—N1	113.09 (15)	C7—C12—H12	119.8
N2—C2—C3	121.51 (16)	C15—N5—C14	109.18 (14)
N2—C2—C5	116.91 (15)	C15—N5—C13	109.49 (13)
C3—C2—C5	121.58 (16)	C14—N5—C13	109.06 (13)
C2—C3—C4	118.22 (16)	N5—C13—C15 ⁱ	109.61 (13)
C2—C3—H3	120.9	N5—C13—H13A	109.7
C4—C3—H3	120.9	C15 ⁱ —C13—H13A	109.7
N3—C4—C3	120.97 (16)	N5—C13—H13B	109.7
N3—C4—C6	116.90 (16)	C15 ⁱ —C13—H13B	109.7
C3—C4—C6	122.13 (16)	H13A—C13—H13B	108.2
C2—C5—H5A	109.5	N5—C14—C14 ⁱ	109.52 (9)
C2—C5—H5B	109.5	N5—C14—H14A	109.8
H5A—C5—H5B	109.5	C14 ⁱ —C14—H14A	109.8
C2—C5—H5C	109.5	N5—C14—H14B	109.8
H5A—C5—H5C	109.5	C14 ⁱ —C14—H14B	109.8
H5B—C5—H5C	109.5	H14A—C14—H14B	108.2
C4—C6—H6A	109.5	N5—C15—C13 ⁱ	109.84 (13)
C4—C6—H6B	109.5	N5—C15—H15A	109.7

H6A—C6—H6B	109.5	C13 ⁱ —C15—H15A	109.7
C4—C6—H6C	109.5	N5—C15—H15B	109.7
H6A—C6—H6C	109.5	C13 ⁱ —C15—H15B	109.7
H6B—C6—H6C	109.5	H15A—C15—H15B	108.2
C12—C7—C8	119.89 (15)		
O2—S1—N1—C1	-66.25 (15)	O2—S1—C7—C8	-166.16 (13)
O1—S1—N1—C1	167.54 (13)	O1—S1—C7—C8	-38.28 (16)
C7—S1—N1—C1	52.51 (15)	N1—S1—C7—C8	72.86 (15)
C2—N2—C1—N3	1.0 (3)	C12—C7—C8—C9	-1.4 (3)
C2—N2—C1—N1	-179.64 (15)	S1—C7—C8—C9	-177.55 (13)
C4—N3—C1—N2	-1.6 (3)	C7—C8—C9—C10	1.0 (3)
C4—N3—C1—N1	179.01 (15)	C8—C9—C10—N4	-177.89 (17)
S1—N1—C1—N2	28.3 (2)	C8—C9—C10—C11	0.4 (3)
S1—N1—C1—N3	-152.26 (12)	N4—C10—C11—C12	176.87 (18)
C1—N2—C2—C3	0.3 (2)	C9—C10—C11—C12	-1.5 (3)
C1—N2—C2—C5	-179.32 (15)	C10—C11—C12—C7	1.1 (3)
N2—C2—C3—C4	-1.0 (3)	C8—C7—C12—C11	0.3 (3)
C5—C2—C3—C4	178.66 (16)	S1—C7—C12—C11	176.48 (15)
C1—N3—C4—C3	0.8 (2)	C15—N5—C13—C15 ⁱ	-61.24 (15)
C1—N3—C4—C6	-178.58 (15)	C14—N5—C13—C15 ⁱ	58.17 (18)
C2—C3—C4—N3	0.4 (3)	C15—N5—C14—C14 ⁱ	57.7 (2)
C2—C3—C4—C6	179.74 (16)	C13—N5—C14—C14 ⁱ	-61.9 (2)
O2—S1—C7—C12	17.69 (17)	C14—N5—C15—C13 ⁱ	-61.68 (17)
O1—S1—C7—C12	145.57 (14)	C13—N5—C15—C13 ⁱ	57.66 (16)
N1—S1—C7—C12	-103.29 (15)		

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N \cdots N5 ⁱⁱ	0.88 (2)	1.90 (2)	2.768 (2)	169 (2)
N4—H2N \cdots O2 ⁱⁱⁱ	0.88 (1)	2.48 (2)	3.058 (2)	124 (2)
N4—H2N \cdots N2 ⁱⁱⁱ	0.88 (1)	2.59 (2)	3.376 (2)	149 (2)
N4—H3N \cdots O1 ^{iv}	0.88 (2)	2.15 (2)	3.032 (2)	178 (2)

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