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(E)-4-Amino-N-(1,2-dihydropyridin-2-ylidene)benzenesulfonamide nitromethane monosolvate

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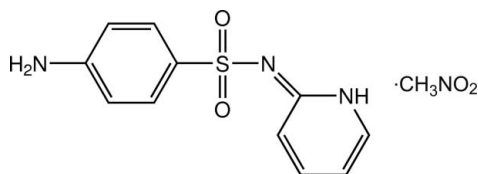
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.045; wR factor = 0.144; data-to-parameter ratio = 14.3.

In the title solvate, $\text{C}_{11}\text{H}_{11}\text{N}_3\text{O}_2\text{S}\cdot\text{CH}_3\text{NO}_2$, the dihedral angle between the benzene ring and the N-containing ring is $85.94(11)^\circ$, and an approximate V shape arises for the sulfonamide molecule. In the crystal, $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds and weak $\text{C}-\text{H}\cdots\text{O}$ interactions link the sulfonamide molecules into a three-dimensional network. The nitromethane solvent molecules are located in the interstitial sites in the sulfonamide network.

Related literature

For background to the applications of sulfonamide compounds, see: Ghorab *et al.* (2009);

**Experimental***Crystal data*

$\text{C}_{11}\text{H}_{11}\text{N}_3\text{O}_2\text{S}\cdot\text{CH}_3\text{NO}_2$ $a = 10.5179(3)$ Å
 $M_r = 310.34$ $b = 12.4857(3)$ Å
 Orthorhombic, *Pbca* $c = 22.7237(6)$ Å

‡ Thomson Reuters ResearcherID: A-5085-2009.

§ College of Pharmacy (Visiting Professor), King Saud University, PO Box 2457, Riyadh 11451, Saudi Arabia. Thomson Reuters ResearcherID: A-3561-2009.

$V = 2984.15(14)$ Å³
 $Z = 8$
 Cu $K\alpha$ radiation

$\mu = 2.14$ mm⁻¹
 $T = 296$ K
 $0.54 \times 0.43 \times 0.31$ mm

Data collection

Bruker SMART APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2009)
 $T_{\min} = 0.392$, $T_{\max} = 0.554$

14569 measured reflections
 2799 independent reflections
 2386 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.062$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.144$
 $S = 1.07$
 2799 reflections
 196 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.25$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.42$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N}2-\text{H}1\text{N}2\cdots\text{O}2^{\text{i}}$	0.87	2.10	2.971 (3)	174
$\text{N}2-\text{H}2\text{N}2\cdots\text{O}1^{\text{ii}}$	0.87	2.34	3.162 (3)	157
$\text{N}3-\text{H}1\text{N}3\cdots\text{N}1^{\text{iii}}$	0.86 (2)	2.09 (2)	2.948 (2)	178 (2)
$\text{C}8-\text{H}8\text{A}\cdots\text{O}1^{\text{iii}}$	0.93	2.52	3.160 (3)	126

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINTE* (Bruker, 2009); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6660).

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 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
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(*E*)-4-Amino-*N*-(1,2-dihydropyridin-2-ylidene)benzenesulfonamide nitromethane monosolvate

Mostafa M. Ghorab, Mansour S. Al-Said, Hazem A. Ghabbour, Suchada Chantrapromma and Hoong-Kun Fun

S1. Comment

As part of our ongoing studies of sulfonamides with potential biological activities (Ghorab *et al.* (2009), the present investigation deals with the synthesis of the title compound, (I), (Fig. 1) a sulfonamide bearing a pyridine moiety to be evaluated as anticancer agent.

The environment of S atom is distorted tetrahedral geometry [angles around S atom are 104.49 (9) - 115.82 (10)°] with two O atoms, one N atom of the amide group and one C atom of the benzene ring. The dihedral angle between the benzene ring and the N atom-containing ring is 85.94 (11)°. The amino group is co-planar with its bound benzene ring with r.m.s. 0.0126 (2) Å for the seven non H atoms (C1–C6/N2).

In the crystal (Fig. 2), the molecules of sulfonamide derivative are linked by N—H···O and N—H···N hydrogen bonds and weak C···H···O interactions (Table 1) into a three dimensional network. The nitromethane solvent molecules are located in the interstitial sites of the sulfonamide network.

S2. Experimental

A mixture of (*E*)-3-(dimethylamino)-1-(thiophen-2-yl)prop-2-en-1-one (1.81 g, 0.01 mole) and sulfapyridine (2.49 g, 0.01 mole) in absolute ethanol (30 ml) was heated under reflux for 12 hr. The reaction mixture was filtered and pink blocks of (I) were obtained by the slow evaporation of an ethanol/nitromethane (3:1) solution at room temperature.

S3. Refinement

Amide H atom was located in a difference map and refined isotropically. The remaining H atoms were placed in calculated positions with $d(\text{N-H}) = 0.87$ Å, $d(\text{C-H}) = 0.93$ for aromatic, and 0.96 Å for CH₃ atoms. The U_{iso} values were constrained to be $1.5U_{\text{eq}}$ of the carrier atom for methyl H atoms and $1.2U_{\text{eq}}$ for the remaining H atoms. A rotating group model was used for the methyl groups.

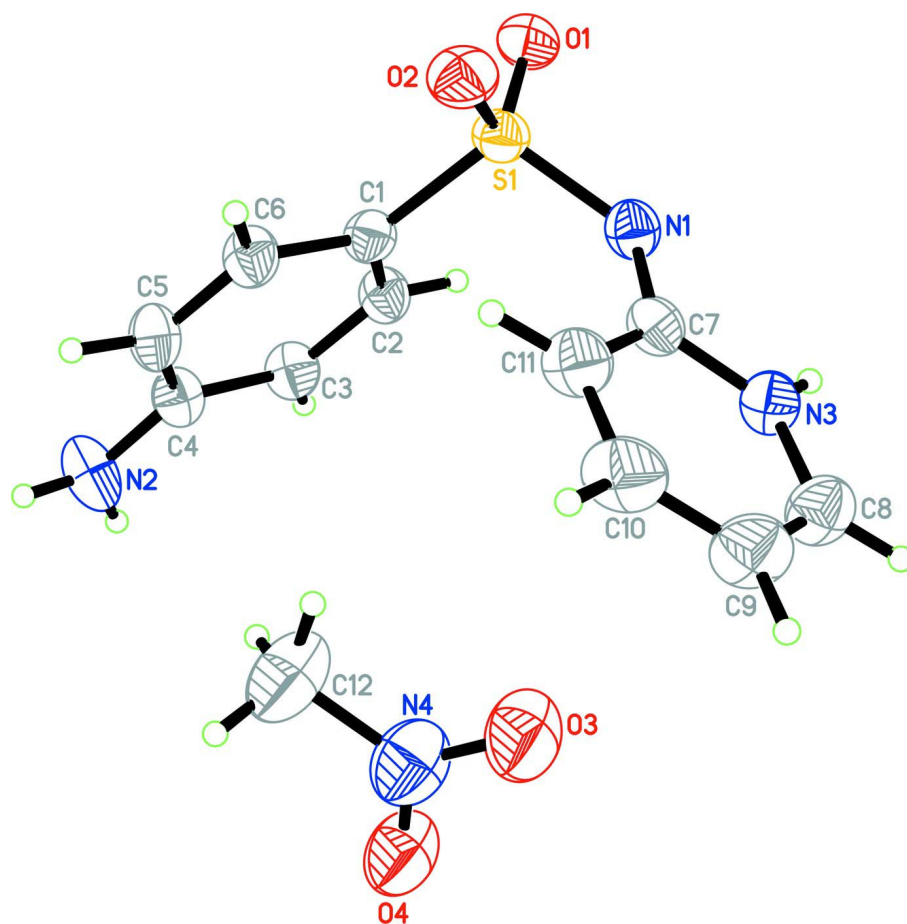


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids.

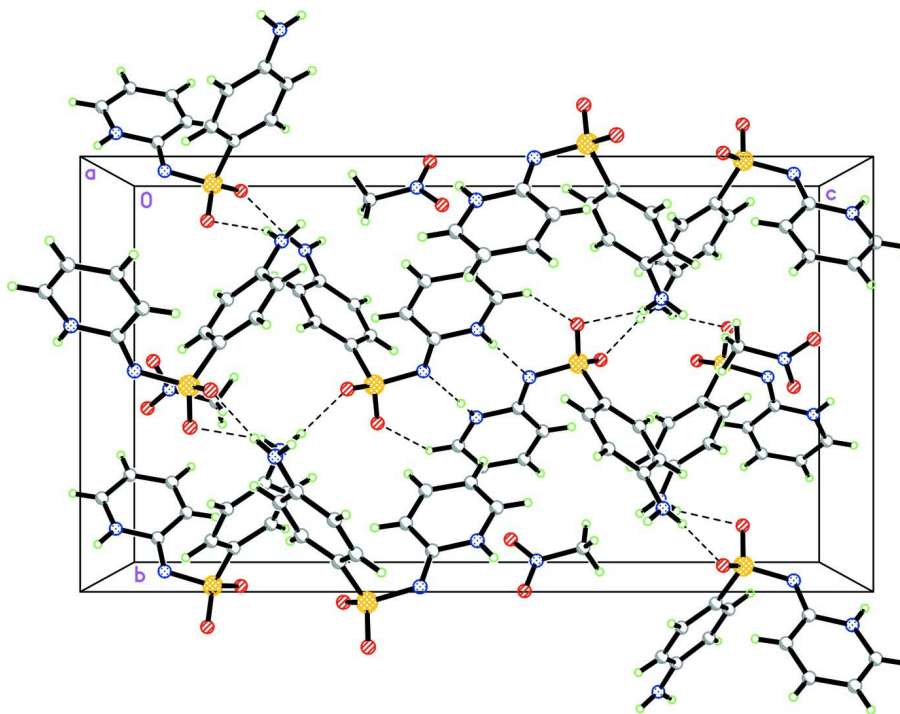


Figure 2

The crystal packing of (I) viewed along the a axis, showing three dimensional network.

(*E*)-4-Amino-*N*-(1,2-dihydropyridin-2-ylidene)benzenesulfonamide nitromethane monosolvate

Crystal data

$C_{11}H_{11}N_3O_2S \cdot CH_3NO_2$

$M_r = 310.34$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 10.5179$ (3) Å

$b = 12.4857$ (3) Å

$c = 22.7237$ (6) Å

$V = 2984.15$ (14) Å³

$Z = 8$

$F(000) = 1296$

$D_x = 1.381$ Mg m⁻³

Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å

Cell parameters from 2799 reflections

$\theta = 5.7\text{--}71.6^\circ$

$\mu = 2.14$ mm⁻¹

$T = 296$ K

Block, pink

$0.54 \times 0.43 \times 0.31$ mm

Data collection

Bruker SMART APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2009)

$T_{\min} = 0.392$, $T_{\max} = 0.554$

14569 measured reflections

2799 independent reflections

2386 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.062$

$\theta_{\max} = 71.6^\circ$, $\theta_{\min} = 5.7^\circ$

$h = -11 \rightarrow 10$

$k = -15 \rightarrow 15$

$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.144$
 $S = 1.07$
 2799 reflections
 196 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
 $w = 1/[\sigma^2(F_o^2) + (0.0843P)^2 + 0.5181P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.25 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.42 \text{ e } \text{\AA}^{-3}$
 Extinction correction: *SHELXTL* (Sheldrick,
 2008), $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0022 (3)

Special details

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.03202 (5)	0.52678 (4)	0.13912 (2)	0.0537 (2)
O1	-0.04470 (16)	0.62229 (11)	0.13586 (7)	0.0664 (4)
O2	0.14832 (16)	0.53613 (12)	0.17203 (7)	0.0696 (4)
N1	0.05275 (16)	0.49231 (13)	0.07212 (8)	0.0549 (4)
N2	-0.27661 (19)	0.18108 (17)	0.24298 (10)	0.0806 (6)
H1N2	-0.2408	0.1417	0.2699	0.097*
H2N2	-0.3364	0.1534	0.2213	0.097*
N3	0.13565 (17)	0.39072 (14)	-0.00078 (8)	0.0587 (4)
H1N3	0.082 (2)	0.4266 (17)	-0.0216 (10)	0.054 (6)*
C1	-0.05963 (18)	0.42658 (14)	0.17206 (8)	0.0499 (4)
C2	-0.1786 (2)	0.40208 (16)	0.14851 (9)	0.0547 (5)
H2A	-0.2093	0.4405	0.1165	0.066*
C3	-0.2504 (2)	0.32171 (17)	0.17237 (9)	0.0576 (5)
H3A	-0.3299	0.3063	0.1565	0.069*
C4	-0.20549 (19)	0.26212 (16)	0.22053 (9)	0.0567 (5)
C5	-0.0891 (2)	0.29027 (18)	0.24482 (9)	0.0628 (5)
H5A	-0.0599	0.2542	0.2780	0.075*
C6	-0.0163 (2)	0.37042 (17)	0.22084 (9)	0.0576 (5)
H6A	0.0623	0.3871	0.2373	0.069*
C7	0.13465 (18)	0.41433 (15)	0.05728 (9)	0.0530 (4)
C8	0.2136 (2)	0.3168 (2)	-0.02582 (12)	0.0741 (6)
H8A	0.2104	0.3049	-0.0662	0.089*
C9	0.2955 (3)	0.2609 (2)	0.00786 (14)	0.0855 (8)

H9A	0.3498	0.2106	-0.0088	0.103*
C10	0.2968 (3)	0.2799 (2)	0.06752 (14)	0.0857 (8)
H10A	0.3523	0.2413	0.0913	0.103*
C11	0.2185 (2)	0.35419 (18)	0.09265 (11)	0.0708 (6)
H11A	0.2205	0.3651	0.1331	0.085*
O3	0.0798 (3)	0.0856 (2)	0.04675 (11)	0.1239 (9)
O4	-0.0699 (2)	-0.0317 (2)	0.06434 (11)	0.1120 (8)
N4	0.0045 (3)	0.0378 (3)	0.08153 (15)	0.1093 (9)
C12	0.0102 (4)	0.0684 (4)	0.14512 (17)	0.1265 (14)
H12A	-0.0722	0.0921	0.1578	0.190*
H12B	0.0706	0.1253	0.1503	0.190*
H12C	0.0358	0.0076	0.1681	0.190*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0566 (4)	0.0492 (3)	0.0551 (3)	-0.00291 (18)	-0.00473 (19)	-0.00030 (17)
O1	0.0793 (11)	0.0487 (8)	0.0712 (10)	0.0061 (7)	0.0029 (7)	0.0001 (6)
O2	0.0653 (10)	0.0710 (9)	0.0724 (10)	-0.0133 (7)	-0.0152 (8)	-0.0063 (7)
N1	0.0541 (9)	0.0559 (8)	0.0546 (9)	0.0045 (7)	-0.0008 (7)	0.0066 (7)
N2	0.0624 (12)	0.0869 (13)	0.0923 (15)	-0.0077 (10)	-0.0068 (10)	0.0371 (11)
N3	0.0544 (10)	0.0631 (9)	0.0587 (10)	0.0083 (8)	-0.0006 (8)	0.0047 (8)
C1	0.0513 (10)	0.0518 (9)	0.0464 (9)	0.0002 (8)	-0.0059 (7)	-0.0004 (7)
C2	0.0549 (11)	0.0588 (10)	0.0505 (10)	0.0017 (8)	-0.0082 (8)	0.0071 (8)
C3	0.0479 (10)	0.0659 (11)	0.0589 (11)	-0.0003 (8)	-0.0077 (8)	0.0038 (9)
C4	0.0494 (10)	0.0626 (10)	0.0580 (11)	0.0039 (9)	0.0040 (8)	0.0082 (8)
C5	0.0618 (13)	0.0740 (12)	0.0525 (10)	0.0043 (10)	-0.0075 (9)	0.0153 (9)
C6	0.0546 (11)	0.0663 (11)	0.0518 (11)	-0.0017 (9)	-0.0110 (8)	0.0050 (8)
C7	0.0476 (10)	0.0520 (9)	0.0595 (11)	-0.0021 (8)	-0.0016 (8)	0.0072 (8)
C8	0.0688 (14)	0.0791 (14)	0.0744 (14)	0.0156 (12)	0.0052 (11)	-0.0043 (11)
C9	0.0784 (17)	0.0795 (15)	0.0985 (19)	0.0293 (14)	0.0006 (14)	-0.0035 (14)
C10	0.0832 (17)	0.0737 (14)	0.100 (2)	0.0258 (13)	-0.0213 (15)	0.0060 (13)
C11	0.0739 (15)	0.0672 (12)	0.0712 (14)	0.0110 (11)	-0.0150 (11)	0.0046 (10)
O3	0.1205 (18)	0.148 (2)	0.1029 (17)	-0.0638 (17)	0.0212 (14)	-0.0115 (15)
O4	0.0996 (15)	0.141 (2)	0.0955 (15)	-0.0502 (15)	0.0236 (13)	-0.0211 (13)
N4	0.0867 (17)	0.139 (2)	0.103 (2)	-0.0042 (17)	0.0109 (16)	-0.0112 (17)
C12	0.122 (3)	0.169 (4)	0.089 (2)	-0.013 (3)	0.004 (2)	-0.033 (2)

Geometric parameters (Å, °)

S1—O2	1.4384 (16)	C5—C6	1.373 (3)
S1—O1	1.4418 (15)	C5—H5A	0.9300
S1—N1	1.5971 (18)	C6—H6A	0.9300
S1—C1	1.7477 (19)	C7—C11	1.410 (3)
N1—C7	1.343 (3)	C8—C9	1.347 (4)
N2—C4	1.358 (3)	C8—H8A	0.9300
N2—H1N2	0.8700	C9—C10	1.377 (4)
N2—H2N2	0.8699	C9—H9A	0.9300

N3—C7	1.352 (3)	C10—C11	1.365 (4)
N3—C8	1.359 (3)	C10—H10A	0.9300
N3—H1N3	0.86 (2)	C11—H11A	0.9300
C1—C6	1.389 (3)	O3—N4	1.268 (4)
C1—C2	1.395 (3)	O4—N4	1.232 (4)
C2—C3	1.368 (3)	N4—C12	1.496 (5)
C2—H2A	0.9300	C12—H12A	0.9600
C3—C4	1.405 (3)	C12—H12B	0.9600
C3—H3A	0.9300	C12—H12C	0.9600
C4—C5	1.388 (3)		
O2—S1—O1	115.82 (10)	C5—C6—C1	120.10 (19)
O2—S1—N1	113.66 (10)	C5—C6—H6A	119.9
O1—S1—N1	104.49 (9)	C1—C6—H6A	119.9
O2—S1—C1	107.73 (10)	N1—C7—N3	114.09 (17)
O1—S1—C1	107.78 (9)	N1—C7—C11	130.1 (2)
N1—S1—C1	106.90 (9)	N3—C7—C11	115.81 (19)
C7—N1—S1	121.49 (14)	C9—C8—N3	120.0 (2)
C4—N2—H1N2	116.5	C9—C8—H8A	120.0
C4—N2—H2N2	118.8	N3—C8—H8A	120.0
H1N2—N2—H2N2	119.1	C8—C9—C10	118.4 (2)
C7—N3—C8	124.15 (19)	C8—C9—H9A	120.8
C7—N3—H1N3	114.7 (15)	C10—C9—H9A	120.8
C8—N3—H1N3	121.1 (16)	C11—C10—C9	121.5 (2)
C6—C1—C2	119.35 (18)	C11—C10—H10A	119.2
C6—C1—S1	121.47 (15)	C9—C10—H10A	119.2
C2—C1—S1	119.17 (15)	C10—C11—C7	120.0 (2)
C3—C2—C1	120.26 (18)	C10—C11—H11A	120.0
C3—C2—H2A	119.9	C7—C11—H11A	120.0
C1—C2—H2A	119.9	O4—N4—O3	122.0 (3)
C2—C3—C4	120.77 (19)	O4—N4—C12	120.8 (3)
C2—C3—H3A	119.6	O3—N4—C12	117.2 (3)
C4—C3—H3A	119.6	N4—C12—H12A	109.5
N2—C4—C5	121.67 (19)	N4—C12—H12B	109.5
N2—C4—C3	120.14 (19)	H12A—C12—H12B	109.5
C5—C4—C3	118.18 (19)	N4—C12—H12C	109.5
C6—C5—C4	121.24 (18)	H12A—C12—H12C	109.5
C6—C5—H5A	119.4	H12B—C12—H12C	109.5
C4—C5—H5A	119.4		
O2—S1—N1—C7	43.60 (19)	C3—C4—C5—C6	-3.3 (3)
O1—S1—N1—C7	170.79 (16)	C4—C5—C6—C1	1.3 (3)
C1—S1—N1—C7	-75.12 (17)	C2—C1—C6—C5	1.2 (3)
O2—S1—C1—C6	-0.9 (2)	S1—C1—C6—C5	-178.33 (17)
O1—S1—C1—C6	-126.50 (18)	S1—N1—C7—N3	176.39 (14)
N1—S1—C1—C6	121.65 (17)	S1—N1—C7—C11	-3.6 (3)
O2—S1—C1—C2	179.59 (16)	C8—N3—C7—N1	178.0 (2)
O1—S1—C1—C2	53.96 (18)	C8—N3—C7—C11	-2.0 (3)

N1—S1—C1—C2	-57.89 (18)	C7—N3—C8—C9	0.8 (4)
C6—C1—C2—C3	-1.6 (3)	N3—C8—C9—C10	0.6 (4)
S1—C1—C2—C3	177.94 (16)	C8—C9—C10—C11	-0.6 (5)
C1—C2—C3—C4	-0.5 (3)	C9—C10—C11—C7	-0.7 (4)
C2—C3—C4—N2	-178.6 (2)	N1—C7—C11—C10	-178.1 (2)
C2—C3—C4—C5	2.9 (3)	N3—C7—C11—C10	1.9 (3)
N2—C4—C5—C6	178.2 (2)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H1N2 \cdots O2 ⁱ	0.87	2.10	2.971 (3)	174
N2—H2N2 \cdots O1 ⁱⁱ	0.87	2.34	3.162 (3)	157
N3—H1N3 \cdots N1 ⁱⁱⁱ	0.86 (2)	2.09 (2)	2.948 (2)	178 (2)
C8—H8A \cdots O1 ⁱⁱⁱ	0.93	2.52	3.160 (3)	126

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