Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

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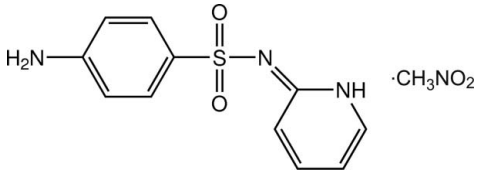
Received 27 February 2012; accepted 6 March 2012

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.144; data-to-parameter ratio = 14.3.

In the title solvate, $C_{11}H_{11}N_3O_2S\cdot CH_3NO_2$, the dihedral angle between the benzene ring and the N-containing ring is 85.94 (11)°, and an approximate V shape arises for the sulfonamide molecule. In the crystal, N—H···O and N—H···N hydrogen bonds and weak C—H···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

$C_{11}H_{11}N_3O_2S\cdot CH_3NO_2$
M_r = 310.34
Orthorhombic, *Pbca*
a = 10.5179 (3) Å
b = 12.4857 (3) Å
c = 22.7237 (6) Å
V = 2984.15 (14) Å^3
Z = 8
Cu $K\alpha$ radiation
μ = 2.14 mm^{-1}
T = 296 K
0.54 × 0.43 × 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_{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
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1*N2*···O2^{i}	0.87	2.10	2.971 (3)	174
N2—H2*N2*···O1^{ii}	0.87	2.34	3.162 (3)	157
N3—H1*N3*···N1^{iii}	0.86 (2)	2.09 (2)	2.948 (2)	178 (2)
C8—H8*A*···O1^{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: *SAINT* (Bruker, 2009); data reduction: *SAINT*; 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).

The authors are grateful for the sponsorship of the Research Center, College of Pharmacy and the Deanship of Scientific Research, King Saud University, Riyadh, Saudi Arabia. HKF and SC thank Universiti Sains Malaysia for the Research University Grant No. 1001/PFIZIK/811160. HKF also thanks the King Saud University, Riyadh, Saudi Arabia, for the award of a visiting Professorship (December 23rd 2011 to January 14th 2012).

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

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Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

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supplementary materials

Acta Cryst. (2012). E**68**, o1030 [doi:10.1107/S1600536812009865]

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

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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.

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.

Refinement

Amide H atom was located in a difference map and refined isotropically. The remaining H atoms were placed in calculated positions with d(N-H) = 0.87 Å, d(C-H) = 0.93 for aromatic, and 0.96 Å for CH_3 atoms. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups.

Computing details

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

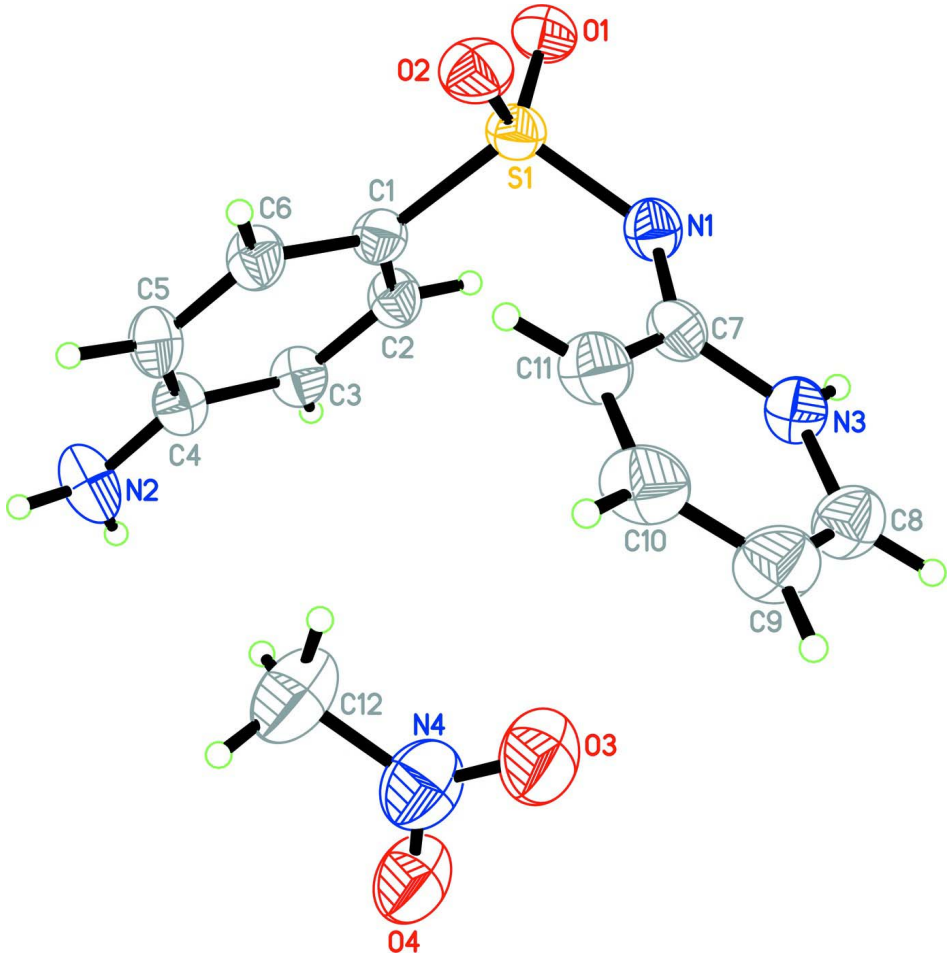

Figure 1

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

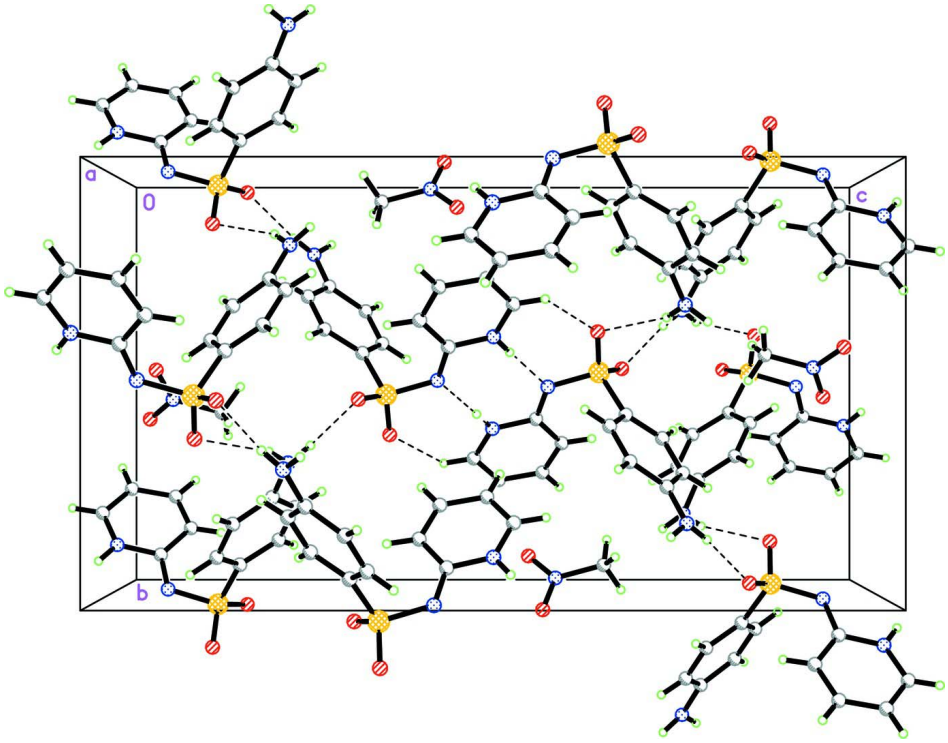

Figure 2

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

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Crystal data

$C_{11}H_{11}N_3O_2S·CH_3NO_2$
$M_r = 310.34$
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) Å^3
$Z = 8$
$F(000) = 1296$
$D_x = 1.381$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 2799 reflections
$\theta = 5.7$–$71.6°$
$\mu = 2.14$ mm^{-1}
$T = 296$ K
Block, pink
0.54 × 0.43 × 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 $I > 2\sigma(I)$
$R_{int} = 0.062$
$\theta_{max} = 71.6°$, $\theta_{min} = 5.7°$
$h = -11 \rightarrow 10$
$k = -15 \rightarrow 15$
$l = -27 \rightarrow 27$

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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{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, º)

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, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1*N*2···O2^{i}	0.87	2.10	2.971 (3)	174
N2—H2*N*2···O1^{ii}	0.87	2.34	3.162 (3)	157
N3—H1*N*3···N1^{iii}	0.86 (2)	2.09 (2)	2.948 (2)	178 (2)
C8—H8*A*···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*.