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1-Ethyl-1*H*-2,1-benzothiazin-4(3*H*)-one 2,2-dioxide

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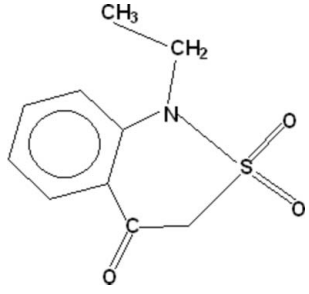
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.108; data-to-parameter ratio = 19.2.

In the title compound, $C_{10}H_{11}NO_3S$, there is distorted tetrahedral geometry around the S atom. The heterocyclic thiazine ring adopts a half-chair conformation. The ethyl and sulfonyl groups form dihedral angles of 82.53 (13) and 88.91 (9)°, respectively, with the plane formed by the benzothiazine ring, excluding the S atom; the S atom and the ethyl group lie on opposite sides of the ring. The molecules are linked into dimers by intermolecular C—H···O hydrogen bonds involving benzene C—H and carbonyl O atoms, thus forming eight-membered rings. The dimers are linked into chains *via* interactions of a similar type. There is an intramolecular C—H···O hydrogen bond.

Related literature

For related literature, see: Hanson *et al.* (1999); Misu & Togo (2003); Shafiq *et al.* (2008); Siddique *et al.* (2006); Siddiqui *et al.* (2007); Tahir *et al.* (2008); Cremer & Pople (1975).



Experimental

Crystal data

$C_{10}H_{11}NO_3S$
M_r = 225.26
Triclinic, $P\overline{1}$
a = 7.0272 (3) Å
b = 8.0448 (4) Å
c = 9.5880 (4) Å
α = 99.124 (3)°
β = 95.075 (3)°
γ = 104.092 (3)°
V = 514.48 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 296 (2) K
0.15 × 0.12 × 0.10 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.965, T_{max} = 0.988
11339 measured reflections
2608 independent reflections
1760 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.107
S = 1.02
2608 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O3^{i}	0.93	2.56	3.478 (3)	169
C8—H8A···O1^{ii}	0.97	2.50	3.388 (3)	152
C9—H9B···O2	0.97	2.36	2.855 (3)	111

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2007); 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, 2003); 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: PV2067).

References

Bruker (2005). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2007). *APEX2* (Version 1.27) and *SAINT* (Version 7.12a). Bruker AXS Inc., Madison, Wisconsin, USA.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Hanson, P. R., Probst, D. A., Robinson, R. E. & Yau, M. (1999). *Tetrahedron Lett.* **40**, 4761–4764.
Misu, Y. & Togo, H. (2003). *Org. Biomol. Chem.* **1**, 1342–1346.
Shafiq, M., Tahir, M. N., Khan, I. U., Siddiqui, W. A. & Arshad, M. N. (2008). *Acta Cryst.* E**64**, o389.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Siddique, W. A., Ahmad, S., Khan, I. U. & Malik, A. (2006). *J. Chem. Soc. Pak.* **28**, 583–589.
Siddiqui, W. A., Ahmad, S., Khan, I. U., Siddiqui, H. L. & Weaver, G. W. (2007). *Synth. Commun.* **37**, 767–773.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Tahir, M. N., Shafiq, M., Khan, I. U., Siddiqui, W. A. & Arshad, M. N. (2008). *Acta Cryst.* E**64**, o557.

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1-Ethyl-1*H*-2,1-benzothiazin-4(3*H*)-one 2,2-dioxide

Muhammad Shafiq, Islam Ullah Khan, M. Nawaz Tahir and Waseeq Ahmad Siddiqui

S1. Comment

Sulfonamides in general, and cyclic sulfonamides (sultams) in particular are important therapeutic compounds (Hanson *et al.*, 1999). Among sultams, 1,2-benzothiazine and 2,1-benzothiazine dioxides (benzosultams) have proven to be biologically active (Misu & Togo, 2003). Due to the importance of 2,1-benzothiazine derivatives in medicinal chemistry, their synthesis has gained enormous attention. After accomplishing the synthesis of a number of 1,2-benzothiazine 1,1-dioxide derivatives (Siddique *et al.*, 2006 and Siddiqui *et al.*, 2007), we have recently started the synthesis of various 2,1-benzothiazine 2,2-dioxide derivatives.

The title compound, (I), was synthesized in continuation to our research on derivatives of 2,1-benzothiazine. It is a cyclized product of methyl 2-(*N*-ethylmethanesulfonamido)benzoate (Shafiq *et al.*, 2008). The hetrocyclic ring adopts a half chair confirmation which may be described by the puckering parameters (Cremer & Pople, 19975): Q = 0.554 (2) Å, θ = 53.6 (2)° and φ = 356.2 (3)°. The structure of (I) can be best compared with its 1-methyl analogue (Tahir *et al.*, 2008). In (I), the bond distance N1—C9 [1.477 (2) Å] is significantly longer than the corresponding distance [1.452 (2) Å] in the 1-methyl analogue. The range of bond angles around S in the two structures are essentially identical. All the atoms in the benzothiazine ring in (I) are nearly planer except that of S1 which is displaced by 0.783 (2) Å from the plane defined by C1—C8/N1, while C9-atom of *N*-ethyl group is at a distance of -0.226 (3) Å. The *N*-ethyl and sulfonyl groups form dihedral angles of 82.53 (13)° and 88.91 (9)°, respectively, with the plane formed by C1—C8/N1 atoms. The dihedral angle between these two groups is 46.66 (5)°. In the asymmetric unit there is an intramolecular H-bond between C9 and O2 atoms. The molecules are dimerized by forming eight member rings through H-bonding between methylene group of thiazine ring and sulfonyl O-atom (C8—H8···O1). The structure is further stabilized by interactions involving phenyl C—H and carbonyl O-atoms (C2—H2···O3) linking dimers into chains. Fig. 2 shows hydrogen bonding interactions; details of H-bonding geometry are given in Table 1.

S2. Experimental

A suspension of hexane-washed sodium hydride (4.6 g, 96.0 mmol., 50% in mineral oil) was prepared in dry dimethylformamide (30 ml). To this suspension, a solution of methyl 2-(*N*-ethylmethanesulfonamido)benzoate (19.02 g, 74.0 mmol) in dry dimethylformamide (70 ml) was added. The reaction mixture was stirred at room temperature (1.5 h) and was poured in a thin stream into hydrochloric acid (3 N, 200 ml). The pH of the mixture was then adjusted to neutral using $NaHCO_3$. After this it was filtered and the filtrate was evaporated under reduced pressure (11 torr) to obtain the title compound (yield; 15 g, 90%); m.p. 354–355 K. Colorless crystals of (I) suitable for X-ray diffraction were grown from MeOH by slow evaporation at room temperature.

S3. Refinement

H atoms were positioned geometrically, with C—H = 0.93, 0.97, and 0.96 Å for aromatic, methylene and methyl H, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C)$, where $x = 1.5$ for methyl H, and $x = 1.2$ for all other H atoms.

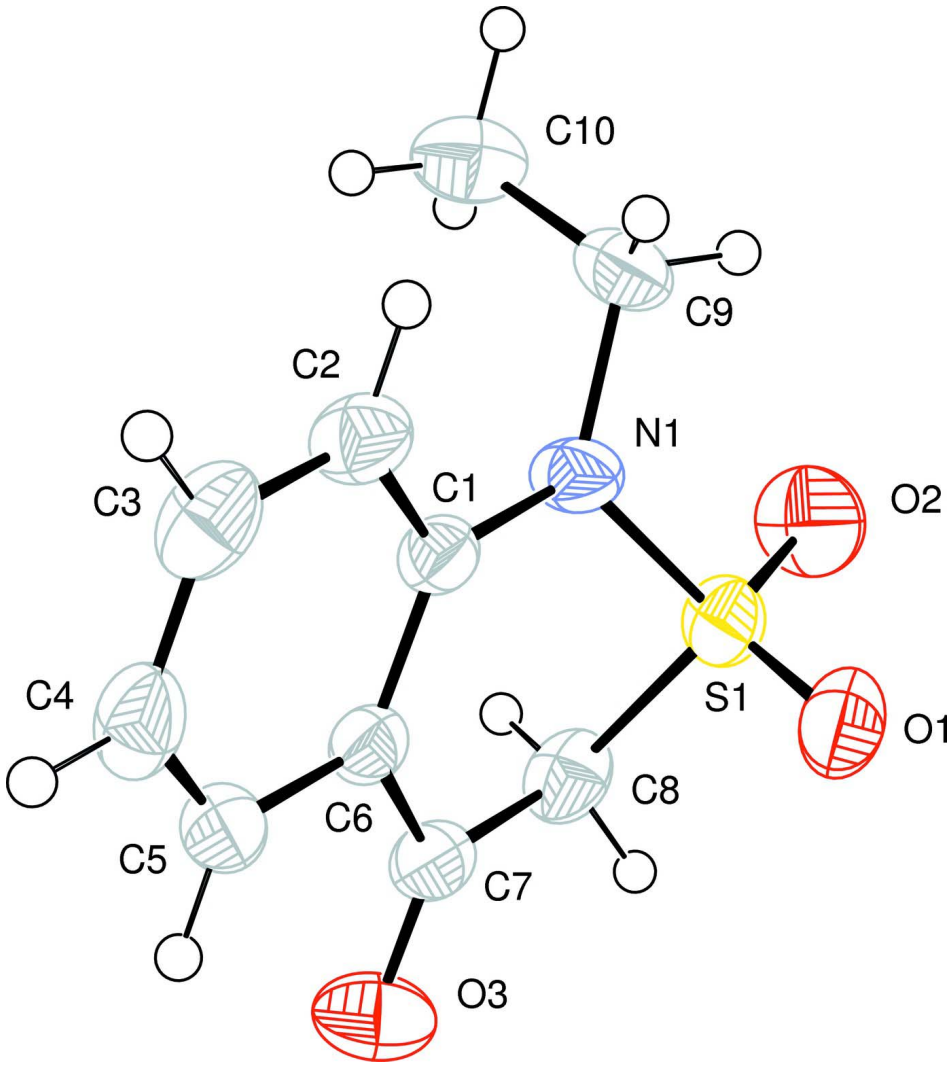

Figure 1

ORTEP-3 (Farrugia, 1997) drawing of (I) with the atom numbering scheme. The thermal ellipsoids are drawn at the 50% probability level.

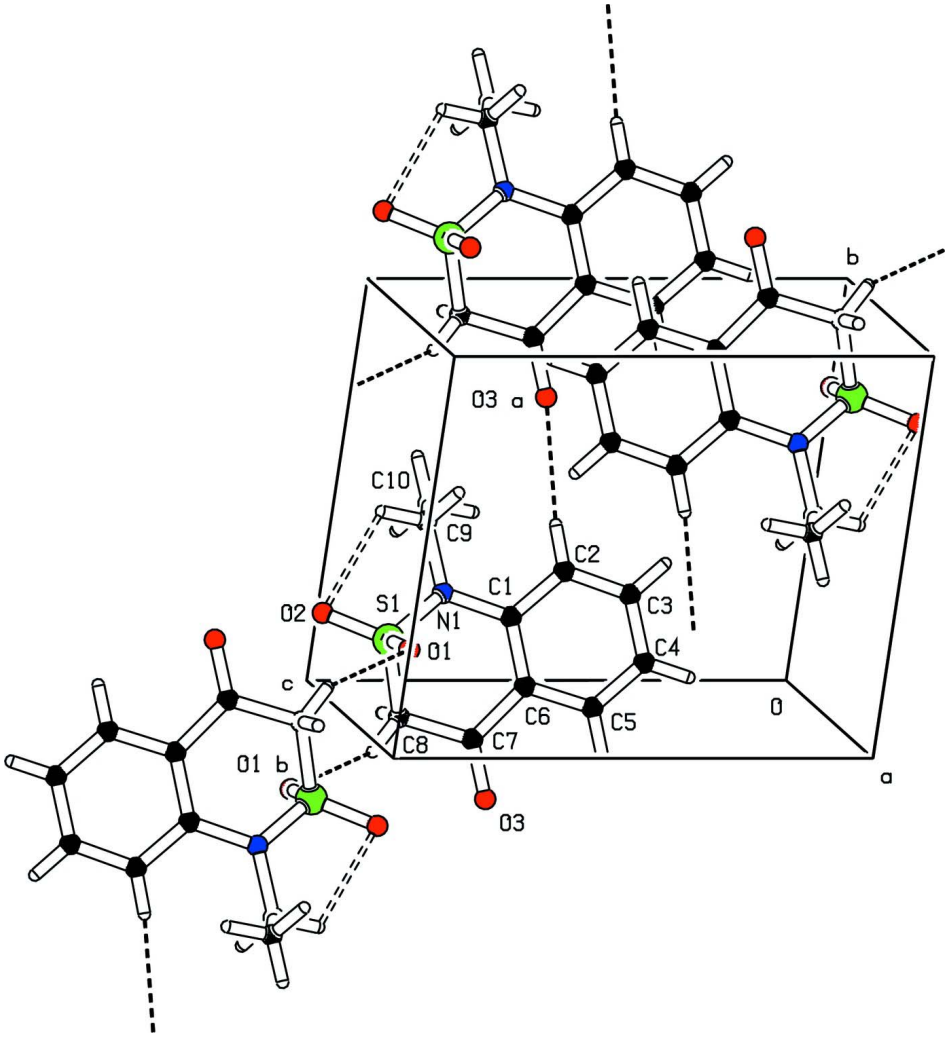

Figure 2

The unit cell packing of (I) (Spek, 2003) showing the intermolecular hydrogen bonds resulting in dimers.

1-Ethyl-1*H*-2,1-benzothiazin-4(3*H*)-one 2,2-dioxide

Crystal data

$C_{10}H_{11}NO_3S$
M_r = 225.26
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 7.0272 (3) Å
b = 8.0448 (4) Å
c = 9.5880 (4) Å
α = 99.124 (3)°
β = 95.075 (3)°
γ = 104.092 (3)°
V = 514.48 (4) Å^3
Z = 2
F(000) = 236
D_x = 1.454 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1760 reflections
θ = 2.2–28.7°
μ = 0.30 mm^{-1}
T = 296 K
Prismatic, colourless
0.15 × 0.12 × 0.10 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 7.40 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.965, T_{max} = 0.988

11339 measured reflections
2608 independent reflections
1760 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 28.7°, θ_{min} = 2.2°
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.107
S = 1.02
2608 reflections
136 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 0.0854P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S1	0.32887 (8)	0.16467 (6)	0.90938 (5)	0.04169 (17)
O1	0.5349 (2)	0.18066 (19)	0.90570 (14)	0.0531 (4)
O2	0.2664 (3)	0.2196 (2)	1.04236 (14)	0.0670 (5)
O3	0.2248 (2)	−0.25167 (19)	0.63616 (17)	0.0632 (4)
N1	0.2390 (2)	0.2638 (2)	0.79263 (15)	0.0415 (4)
C1	0.2558 (3)	0.2075 (2)	0.64688 (17)	0.0328 (4)
C2	0.2726 (3)	0.3249 (3)	0.5533 (2)	0.0429 (5)
H2	0.2746	0.4406	0.5861	0.051*
C3	0.2864 (3)	0.2688 (3)	0.4118 (2)	0.0489 (5)
H3	0.2959	0.3475	0.3498	0.059*
C4	0.2862 (3)	0.1000 (3)	0.3604 (2)	0.0485 (5)
H4	0.2982	0.0651	0.2651	0.058*
C5	0.2684 (3)	−0.0161 (3)	0.45077 (19)	0.0420 (5)
H5	0.2679	−0.1310	0.4160	0.050*
C6	0.2509 (2)	0.0337 (2)	0.59449 (17)	0.0329 (4)
C7	0.2276 (3)	−0.1022 (2)	0.6829 (2)	0.0389 (4)

C8	0.2024 (3)	−0.0522 (3)	0.8374 (2)	0.0445 (5)
H8A	0.2512	−0.1285	0.8919	0.053*
H8B	0.0627	−0.0687	0.8454	0.053*
C9	0.2066 (3)	0.4367 (3)	0.8431 (2)	0.0461 (5)
H9A	0.2951	0.5239	0.8032	0.055*
H9B	0.2378	0.4664	0.9461	0.055*
C10	−0.0036 (3)	0.4394 (3)	0.8011 (3)	0.0575 (6)
H10A	−0.0197	0.5532	0.8354	0.086*
H10B	−0.0914	0.3545	0.8420	0.086*
H10C	−0.0341	0.4120	0.6992	0.086*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0517 (3)	0.0444 (3)	0.0303 (2)	0.0137 (2)	0.00127 (19)	0.01149 (19)
O1	0.0444 (9)	0.0558 (9)	0.0541 (8)	0.0071 (7)	−0.0081 (6)	0.0135 (7)
O2	0.0971 (13)	0.0800 (12)	0.0318 (7)	0.0339 (10)	0.0142 (8)	0.0136 (7)
O3	0.0782 (12)	0.0343 (9)	0.0810 (11)	0.0192 (8)	0.0145 (9)	0.0127 (7)
N1	0.0633 (11)	0.0387 (9)	0.0299 (7)	0.0260 (8)	0.0075 (7)	0.0082 (6)
C1	0.0347 (10)	0.0366 (10)	0.0302 (8)	0.0141 (8)	0.0044 (7)	0.0083 (7)
C2	0.0504 (12)	0.0408 (11)	0.0434 (10)	0.0167 (9)	0.0083 (9)	0.0163 (8)
C3	0.0492 (13)	0.0673 (15)	0.0394 (10)	0.0197 (11)	0.0106 (9)	0.0273 (10)
C4	0.0436 (12)	0.0748 (16)	0.0305 (9)	0.0224 (10)	0.0055 (8)	0.0083 (9)
C5	0.0374 (11)	0.0459 (12)	0.0407 (10)	0.0152 (9)	0.0000 (8)	−0.0022 (8)
C6	0.0297 (9)	0.0366 (11)	0.0337 (9)	0.0118 (8)	0.0026 (7)	0.0066 (7)
C7	0.0332 (10)	0.0337 (11)	0.0498 (11)	0.0092 (8)	0.0013 (8)	0.0095 (8)
C8	0.0446 (12)	0.0431 (12)	0.0485 (11)	0.0080 (9)	0.0024 (9)	0.0237 (9)
C9	0.0618 (14)	0.0334 (11)	0.0423 (10)	0.0152 (10)	0.0087 (9)	−0.0004 (8)
C10	0.0615 (15)	0.0474 (14)	0.0693 (14)	0.0239 (11)	0.0180 (11)	0.0075 (11)

Geometric parameters (Å, º)

S1—O2	1.4244 (14)	C4—H4	0.9300
S1—O1	1.4260 (14)	C5—C6	1.398 (2)
S1—N1	1.6405 (15)	C5—H5	0.9300
S1—C8	1.750 (2)	C6—C7	1.473 (2)
O3—C7	1.210 (2)	C7—C8	1.510 (3)
N1—C1	1.424 (2)	C8—H8A	0.9700
N1—C9	1.477 (2)	C8—H8B	0.9700
C1—C2	1.395 (2)	C9—C10	1.503 (3)
C1—C6	1.400 (2)	C9—H9A	0.9700
C2—C3	1.380 (3)	C9—H9B	0.9700
C2—H2	0.9300	C10—H10A	0.9600
C3—C4	1.368 (3)	C10—H10B	0.9600
C3—H3	0.9300	C10—H10C	0.9600
C4—C5	1.363 (3)		
O2—S1—O1	118.03 (10)	C5—C6—C1	119.07 (16)

O2—S1—N1	107.40 (9)	C5—C6—C7	117.38 (17)
O1—S1—N1	111.71 (9)	C1—C6—C7	123.54 (16)
O2—S1—C8	110.60 (10)	O3—C7—C6	122.76 (18)
O1—S1—C8	107.58 (9)	O3—C7—C8	119.04 (17)
N1—S1—C8	100.05 (8)	C6—C7—C8	118.20 (16)
C1—N1—C9	121.18 (14)	C7—C8—S1	112.13 (13)
C1—N1—S1	117.15 (12)	C7—C8—H8A	109.2
C9—N1—S1	118.59 (12)	S1—C8—H8A	109.2
C2—C1—C6	119.03 (16)	C7—C8—H8B	109.2
C2—C1—N1	120.08 (16)	S1—C8—H8B	109.2
C6—C1—N1	120.87 (15)	H8A—C8—H8B	107.9
C3—C2—C1	119.70 (18)	N1—C9—C10	111.54 (17)
C3—C2—H2	120.2	N1—C9—H9A	109.3
C1—C2—H2	120.2	C10—C9—H9A	109.3
C4—C3—C2	121.64 (18)	N1—C9—H9B	109.3
C4—C3—H3	119.2	C10—C9—H9B	109.3
C2—C3—H3	119.2	H9A—C9—H9B	108.0
C5—C4—C3	119.13 (18)	C9—C10—H10A	109.5
C5—C4—H4	120.4	C9—C10—H10B	109.5
C3—C4—H4	120.4	H10A—C10—H10B	109.5
C4—C5—C6	121.41 (18)	C9—C10—H10C	109.5
C4—C5—H5	119.3	H10A—C10—H10C	109.5
C6—C5—H5	119.3	H10B—C10—H10C	109.5

O2—S1—N1—C1	−170.18 (14)	C4—C5—C6—C7	178.61 (17)
O1—S1—N1—C1	58.92 (16)	C2—C1—C6—C5	1.7 (3)
C8—S1—N1—C1	−54.70 (16)	N1—C1—C6—C5	−179.84 (16)
O2—S1—N1—C9	29.41 (18)	C2—C1—C6—C7	−178.16 (17)
O1—S1—N1—C9	−101.49 (15)	N1—C1—C6—C7	0.3 (3)
C8—S1—N1—C9	144.89 (15)	C5—C6—C7—O3	0.5 (3)
C9—N1—C1—C2	9.9 (3)	C1—C6—C7—O3	−179.56 (17)
S1—N1—C1—C2	−149.95 (15)	C5—C6—C7—C8	−178.23 (15)
C9—N1—C1—C6	−168.48 (17)	C1—C6—C7—C8	1.7 (3)
S1—N1—C1—C6	31.6 (2)	O3—C7—C8—S1	149.47 (16)
C6—C1—C2—C3	−0.7 (3)	C6—C7—C8—S1	−31.7 (2)
N1—C1—C2—C3	−179.18 (17)	O2—S1—C8—C7	166.56 (13)
C1—C2—C3—C4	−0.8 (3)	O1—S1—C8—C7	−63.23 (15)
C2—C3—C4—C5	1.2 (3)	N1—S1—C8—C7	53.53 (15)
C3—C4—C5—C6	−0.2 (3)	C1—N1—C9—C10	75.8 (2)
C4—C5—C6—C1	−1.3 (3)	S1—N1—C9—C10	−124.64 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O3^{i}	0.93	2.56	3.478 (3)	169

C8—H8*A*···O1^{ii}	0.97	2.50	3.388 (3)	152
C9—H9*B*···O2	0.97	2.36	2.855 (3)	111

Symmetry codes: (i) x, y+1, z; (ii) −x+1, −y, −z+2.