

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

N-(3-Chlorophenyl)-1,2-benzisothiazol-3-amine 1,1-dioxide

Tariq Saeed Shah,^a Waseeq Ahmad Siddiqui,^a M. Nawaz Tahir^{b*} and Ghulam Hussain^a

^aDepartment of Chemistry, University of Sargodha, Sargodha, Pakistan, and^bDepartment of Physics, University of Sargodha, Sargodha, Pakistan

Correspondence e-mail: dmntahir_uos@yahoo.com

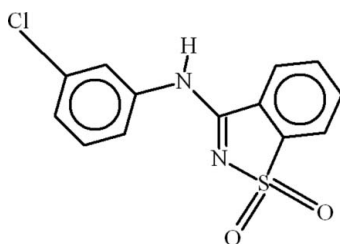
Received 31 March 2010; accepted 25 April 2010

Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.056; wR factor = 0.115; data-to-parameter ratio = 15.7.

In the title compound, $\text{C}_{13}\text{H}_9\text{ClN}_2\text{O}_2\text{S}$, the dihedral angle between the aromatic ring systems is 6.00 (12)° and an intramolecular $\text{C}-\text{H}\cdots\text{N}$ interaction generates an $S(6)$ ring. In the crystal, molecules interact by way of $\text{C}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ bonds, generating $R_2^1(7)$ and $R_2^2(10)$ ring motifs, and aromatic $\pi-\pi$ stacking interactions [centroid-centroid separations = 3.730 (3) and 3.733 (2) Å] help to consolidate the packing.

Related literature

For other saccharin derivatives, see: Rafique *et al.* (2009); Siddiqui *et al.* (2010). For a related structure, see: Brigas *et al.* (2001). For graph-set theory, see: Bernstein *et al.* (1995).



Experimental

Crystal data

 $\text{C}_{13}\text{H}_9\text{ClN}_2\text{O}_2\text{S}$ $M_r = 292.73$ Triclinic, $P\bar{1}$ $a = 7.2223$ (10) Å $b = 7.9138$ (12) Å $c = 11.2175$ (17) Å $\alpha = 96.178$ (6)° $\beta = 98.840$ (5)° $\gamma = 97.574$ (5)° $V = 622.63$ (16) Å³ $Z = 2$ Mo $K\alpha$ radiation $\mu = 0.47$ mm⁻¹
 $T = 296$ K $0.28 \times 0.10 \times 0.08$ mm

Data collection

Bruker Kappa APEXII CCD diffractometer

Absorption correction: multi-scan (SADABS; Bruker, 2005)
 $T_{\min} = 0.947$, $T_{\max} = 0.962$ 10481 measured reflections
2700 independent reflections
1244 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.088$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.056$ $wR(F^2) = 0.115$ $S = 0.98$

2700 reflections

172 parameters

H-atom parameters constrained

 $\Delta\rho_{\max} = 0.25$ e Å⁻³ $\Delta\rho_{\min} = -0.26$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C13}-\text{H13}\cdots\text{N2}$	0.93	2.28	2.901 (5)	124
$\text{N1}-\text{H1}\cdots\text{O1}^i$	0.86	2.24	3.084 (4)	165
$\text{C5}-\text{H5}\cdots\text{O1}^i$	0.93	2.51	3.396 (4)	159
$\text{C2}-\text{H2}\cdots\text{O2}^{ii}$	0.93	2.42	3.302 (4)	158

Symmetry codes: (i) $x - 1, y, z$; (ii) $-x + 3, -y + 1, -z + 1$.

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; 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, 2009); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON.

The authors acknowledge the provision of funds for the purchase of diffractometer and encouragement by Dr Muhammad Akram Chaudhary, Vice Chancellor, University of Sargodha. The authors also acknowledge technical support provided by Bana International, Karachi, Pakistan.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5389).

References

- Bernstein, J., Davis, R. E., Shimon, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Brigas, A. F., Clegg, W., Dillon, C. J., Fonseca, C. F. C. & Johnstone, R. A. W. (2001). *J. Chem. Soc. Perkin Trans. 2*, pp. 1315–1324.
- Bruker (2005). SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2007). APEX2 and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Rafique, M., Hussain, G., Siddiqui, W. A. & Tahir, M. N. (2009). *Acta Cryst. E65*, o1883.
- Sheldrick, G. M. (2008). *Acta Cryst. A64*, 112–122.
- Siddiqui, W. A., Ahmad, S., Siddiqui, H. L., Hussain, T. & Parvez, M. (2010). *J. Chem. Crystallogr.* **40**, 116–121.
- Spek, A. L. (2009). *Acta Cryst. D65*, 148–155.

supporting information

Acta Cryst. (2010). E66, o1220 [https://doi.org/10.1107/S1600536810015163]

N*-(3-Chlorophenyl)-1,2-benzisothiazol-3-amine 1,1-dioxide*Tariq Saeed Shah, Waseeq Ahmad Siddiqui, M. Nawaz Tahir and Ghulam Hussain****S1. Comment**

Due to the interest in obtaining new derivatives of saccharin (Rafique *et al.*, 2009; Siddiqui *et al.*, 2010), we wish to report the preparation and crystal structure of the title compound (I, Fig. 1).

The crystal structure of (II) *N*-(1,1-Dioxo-1,2-benzisothiazol-3-yl)-4-methoxyaniline (Brigas *et al.*, 2001) and (III) *N*-(1,1-Dioxo-1,2-benzisothiazol-3-yl)-3-methylaniline (Brigas *et al.*, 2001) have been published. The title compound differs from (II) and (III) due to attachment of chloro substitution on the aniline. In (I), 1,2-benzisothiazol-3-amine A (C7/N2/S1) and 3-Chlorophenyl B (C8–C13/CL1) are planar with maximum r. m. s. deviations of 0.0080 Å and 0.0033 Å from the respective mean square planes. The dihedral angle between A/B is 6.00 (12)°. There exists an intramolecular H-bonding of C–H...N type forming S(6) ring motif (Bernstein *et al.*, 1995). The molecules are stabilized in the form of polymeric sheets due to intermolecular H-bondings (Table 1, Fig. 2) completing R₂¹(7) and R₂²(10) ring motifs. There exist π – π interactions at a distance of 3.733 (2) Å and 3.730 (2) Å, between the centroids of the benzene rings Cg1 (C1–C6) and Cg2 (C8–C13) respectively, [Cg1...Cg1ⁱ: i = 2 - x, -y, 1 - z] and [Cg2...Cg2ⁱⁱ: ii = 1 - x, -y, - z].

S2. Experimental

A mixture of saccharin (1.0 g, 5.46 mmol) and *m*-chloroaniline (5 ml, in excess) was heated to reflux on an oil-bath (4 h), cooled to room temperature and kept overnight in a freezer. The solvent was evaporated under reduced pressure and the brownish yellow paste obtained was washed with benzene (4 × 25 ml) to obtain the bright light brown crystalline product (1.25, 78%, m. p. 578-579 K). Recrystallisation solvent: MeOH:AcOEt (1:1): the solution was subjected to slow evaporation at room temperature to obtain colourless needles of (I).

S3. Refinement

The H-atoms were positioned geometrically (C–H = 0.93, N–H = 0.86 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$.

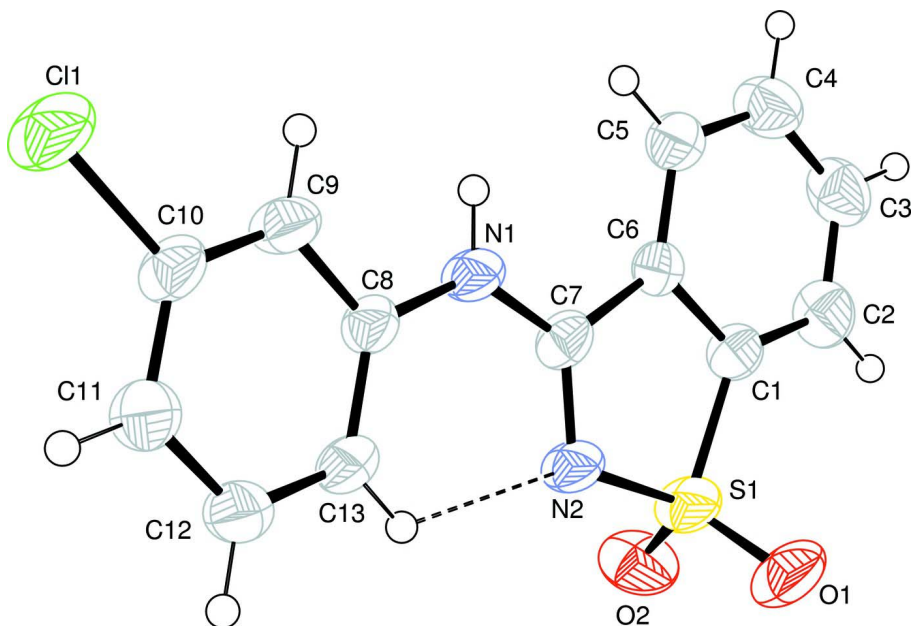


Figure 1

View of (I) with displacement ellipsoids drawn at the 50% probability level.

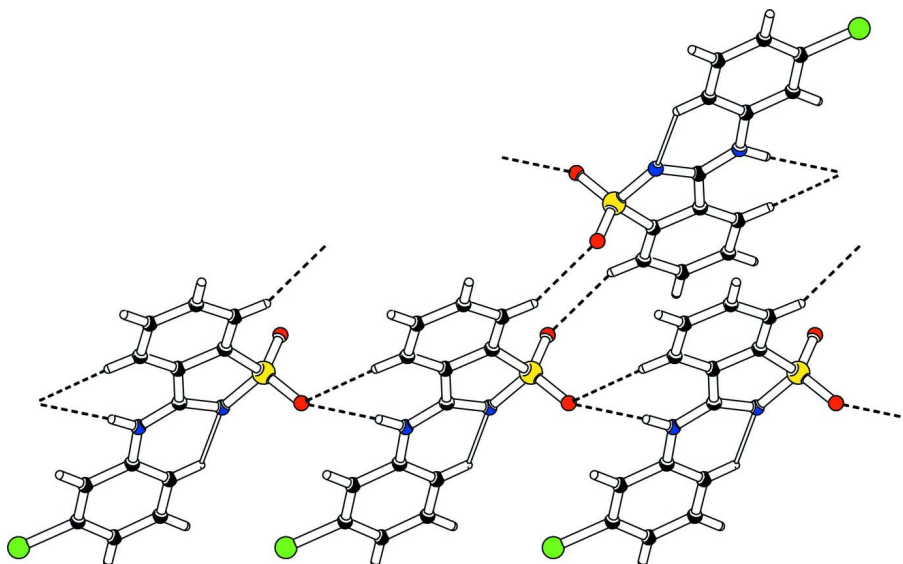


Figure 2

The partial packing of (I), which shows that molecules form polymeric sheets with various ring motifs.

N-(3-Chlorophenyl)-1,2-benzisothiazol-3-amine 1,1-dioxide

Crystal data

$C_{13}H_9ClN_2O_2S$

$M_r = 292.73$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 7.2223$ (10) Å

$b = 7.9138$ (12) Å

$c = 11.2175$ (17) Å

$\alpha = 96.178$ (6)°

$\beta = 98.840$ (5)°

$\gamma = 97.574$ (5)°

$V = 622.63$ (16) Å³

$Z = 2$

$F(000) = 300$
 $D_x = 1.561 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 1244 reflections
 $\theta = 2.6\text{--}27.1^\circ$

$\mu = 0.47 \text{ mm}^{-1}$
 $T = 296 \text{ K}$
 Needle, colourless
 $0.28 \times 0.10 \times 0.08 \text{ mm}$

Data collection

Bruker Kappa APEXII CCD
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 Detector resolution: 7.60 pixels mm^{-1}
 ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2005)
 $T_{\min} = 0.947$, $T_{\max} = 0.962$

10481 measured reflections
 2700 independent reflections
 1244 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.088$
 $\theta_{\max} = 27.1^\circ$, $\theta_{\min} = 2.6^\circ$
 $h = -8 \rightarrow 9$
 $k = -10 \rightarrow 10$
 $l = -14 \rightarrow 14$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.056$
 $wR(F^2) = 0.115$
 $S = 0.98$
 2700 reflections
 172 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
 $w = 1/[\sigma^2(F_o^2) + (0.0363P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.25 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.26 \text{ e \AA}^{-3}$

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 $F^2 > \sigma(F^2)$ 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	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	1.1663 (5)	0.2836 (4)	0.4969 (3)	0.0380 (9)
C2	1.2431 (5)	0.3741 (5)	0.6082 (3)	0.0512 (11)
H2	1.3719	0.4162	0.6278	0.061*
C3	1.1225 (6)	0.4010 (5)	0.6905 (3)	0.0566 (12)
H3	1.1705	0.4625	0.7668	0.068*
C4	0.9329 (6)	0.3379 (5)	0.6609 (4)	0.0557 (11)
H4	0.8542	0.3572	0.7177	0.067*
C5	0.8563 (5)	0.2460 (5)	0.5481 (3)	0.0466 (10)
H5	0.7275	0.2038	0.5289	0.056*
C6	0.9745 (5)	0.2185 (4)	0.4651 (3)	0.0352 (9)
C7	0.9368 (5)	0.1246 (4)	0.3407 (3)	0.0374 (9)

C8	0.6944 (4)	-0.0592 (4)	0.1789 (3)	0.0361 (9)
C9	0.5043 (5)	-0.1312 (5)	0.1558 (3)	0.0426 (10)
H9	0.4257	-0.1074	0.2119	0.051*
C10	0.4338 (5)	-0.2380 (5)	0.0491 (3)	0.0415 (10)
C11	0.5448 (5)	-0.2769 (5)	-0.0357 (3)	0.0482 (10)
H11	0.4944	-0.3497	-0.1073	0.058*
C12	0.7330 (5)	-0.2050 (5)	-0.0116 (3)	0.0485 (11)
H12	0.8109	-0.2302	-0.0677	0.058*
C13	0.8080 (5)	-0.0967 (5)	0.0939 (3)	0.0457 (10)
H13	0.9353	-0.0485	0.1081	0.055*
C11	0.19425 (13)	-0.32624 (14)	0.02129 (9)	0.0658 (4)
N1	0.7608 (4)	0.0472 (4)	0.2912 (3)	0.0417 (8)
H1	0.6751	0.0650	0.3345	0.050*
N2	1.0821 (4)	0.1190 (4)	0.2841 (3)	0.0428 (8)
O1	1.4055 (3)	0.1090 (3)	0.4010 (2)	0.0619 (8)
O2	1.3450 (4)	0.3710 (3)	0.3184 (2)	0.0645 (8)
S1	1.27387 (13)	0.22471 (14)	0.37119 (9)	0.0481 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.029 (2)	0.039 (2)	0.041 (2)	-0.0055 (17)	0.0023 (17)	0.0026 (19)
C2	0.044 (2)	0.052 (3)	0.047 (3)	-0.011 (2)	-0.003 (2)	-0.001 (2)
C3	0.062 (3)	0.062 (3)	0.039 (3)	-0.004 (2)	0.002 (2)	-0.002 (2)
C4	0.056 (3)	0.063 (3)	0.045 (3)	-0.001 (2)	0.013 (2)	-0.001 (2)
C5	0.036 (2)	0.059 (3)	0.041 (2)	-0.0003 (19)	0.0038 (19)	0.002 (2)
C6	0.033 (2)	0.039 (2)	0.033 (2)	0.0038 (17)	0.0032 (17)	0.0035 (19)
C7	0.028 (2)	0.037 (2)	0.043 (2)	-0.0002 (17)	0.0007 (17)	0.0000 (19)
C8	0.028 (2)	0.040 (2)	0.036 (2)	0.0022 (17)	0.0018 (17)	-0.0021 (19)
C9	0.030 (2)	0.048 (3)	0.047 (3)	-0.0001 (18)	0.0107 (18)	-0.005 (2)
C10	0.027 (2)	0.042 (3)	0.049 (3)	-0.0019 (17)	0.0015 (18)	-0.005 (2)
C11	0.040 (2)	0.054 (3)	0.044 (2)	-0.0013 (19)	0.0037 (19)	-0.010 (2)
C12	0.036 (2)	0.064 (3)	0.042 (2)	0.002 (2)	0.0083 (19)	-0.007 (2)
C13	0.024 (2)	0.065 (3)	0.044 (2)	0.0020 (18)	0.0045 (18)	0.001 (2)
C11	0.0325 (6)	0.0807 (9)	0.0706 (8)	-0.0122 (5)	0.0055 (5)	-0.0205 (6)
N1	0.0249 (16)	0.054 (2)	0.0411 (19)	-0.0042 (14)	0.0069 (14)	-0.0036 (16)
N2	0.0248 (16)	0.057 (2)	0.0421 (19)	-0.0024 (14)	0.0065 (14)	-0.0062 (16)
O1	0.0289 (15)	0.077 (2)	0.073 (2)	0.0088 (14)	0.0005 (13)	-0.0092 (17)
O2	0.0539 (18)	0.070 (2)	0.0632 (19)	-0.0227 (15)	0.0210 (15)	0.0020 (16)
S1	0.0261 (5)	0.0609 (8)	0.0494 (7)	-0.0073 (5)	0.0045 (5)	-0.0087 (6)

Geometric parameters (Å, °)

C1—C2	1.367 (5)	C8—C9	1.389 (4)
C1—C6	1.390 (4)	C8—N1	1.417 (4)
C1—S1	1.760 (4)	C9—C10	1.374 (5)
C2—C3	1.382 (5)	C9—H9	0.9300
C2—H2	0.9300	C10—C11	1.370 (5)

C3—C4	1.371 (5)	C10—C11	1.746 (3)
C3—H3	0.9300	C11—C12	1.376 (4)
C4—C5	1.384 (5)	C11—H11	0.9300
C4—H4	0.9300	C12—C13	1.375 (5)
C5—C6	1.376 (4)	C12—H12	0.9300
C5—H5	0.9300	C13—H13	0.9300
C6—C7	1.477 (5)	N1—H1	0.8600
C7—N2	1.310 (4)	N2—S1	1.635 (3)
C7—N1	1.343 (4)	O1—S1	1.432 (3)
C8—C13	1.384 (4)	O2—S1	1.428 (3)
C2—C1—C6	122.5 (3)	C10—C9—H9	120.4
C2—C1—S1	130.4 (3)	C8—C9—H9	120.4
C6—C1—S1	107.1 (3)	C11—C10—C9	122.3 (3)
C1—C2—C3	117.7 (4)	C11—C10—C11	119.2 (3)
C1—C2—H2	121.2	C9—C10—C11	118.5 (3)
C3—C2—H2	121.2	C10—C11—C12	118.0 (3)
C4—C3—C2	120.7 (4)	C10—C11—H11	121.0
C4—C3—H3	119.6	C12—C11—H11	121.0
C2—C3—H3	119.6	C13—C12—C11	121.3 (3)
C3—C4—C5	121.3 (4)	C13—C12—H12	119.3
C3—C4—H4	119.4	C11—C12—H12	119.3
C5—C4—H4	119.4	C12—C13—C8	120.0 (3)
C6—C5—C4	118.7 (4)	C12—C13—H13	120.0
C6—C5—H5	120.7	C8—C13—H13	120.0
C4—C5—H5	120.7	C7—N1—C8	130.1 (3)
C5—C6—C1	119.2 (3)	C7—N1—H1	115.0
C5—C6—C7	131.3 (3)	C8—N1—H1	115.0
C1—C6—C7	109.5 (3)	C7—N2—S1	109.9 (2)
N2—C7—N1	122.9 (3)	O2—S1—O1	115.88 (17)
N2—C7—C6	116.9 (3)	O2—S1—N2	110.41 (16)
N1—C7—C6	120.2 (3)	O1—S1—N2	109.76 (16)
C13—C8—C9	119.3 (3)	O2—S1—C1	111.86 (17)
C13—C8—N1	123.8 (3)	O1—S1—C1	110.64 (17)
C9—C8—N1	116.9 (3)	N2—S1—C1	96.63 (15)
C10—C9—C8	119.1 (3)		
C6—C1—C2—C3	-0.3 (6)	C11—C10—C11—C12	179.7 (3)
S1—C1—C2—C3	-179.8 (3)	C10—C11—C12—C13	-0.3 (6)
C1—C2—C3—C4	0.3 (6)	C11—C12—C13—C8	0.6 (6)
C2—C3—C4—C5	-0.1 (6)	C9—C8—C13—C12	-0.5 (5)
C3—C4—C5—C6	0.0 (6)	N1—C8—C13—C12	178.3 (3)
C4—C5—C6—C1	-0.1 (5)	N2—C7—N1—C8	4.0 (6)
C4—C5—C6—C7	178.8 (4)	C6—C7—N1—C8	-174.8 (3)
C2—C1—C6—C5	0.2 (6)	C13—C8—N1—C7	-3.2 (6)
S1—C1—C6—C5	179.8 (3)	C9—C8—N1—C7	175.6 (3)
C2—C1—C6—C7	-178.9 (3)	N1—C7—N2—S1	-179.4 (3)
S1—C1—C6—C7	0.8 (4)	C6—C7—N2—S1	-0.6 (4)

C5—C6—C7—N2	-179.1 (4)	C7—N2—S1—O2	-115.4 (3)
C1—C6—C7—N2	-0.1 (4)	C7—N2—S1—O1	115.7 (3)
C5—C6—C7—N1	-0.2 (6)	C7—N2—S1—C1	0.9 (3)
C1—C6—C7—N1	178.7 (3)	C2—C1—S1—O2	-66.3 (4)
C13—C8—C9—C10	0.1 (5)	C6—C1—S1—O2	114.1 (3)
N1—C8—C9—C10	-178.8 (3)	C2—C1—S1—O1	64.5 (4)
C8—C9—C10—C11	0.2 (6)	C6—C1—S1—O1	-115.0 (3)
C8—C9—C10—C11	-179.6 (3)	C2—C1—S1—N2	178.6 (4)
C9—C10—C11—C12	-0.1 (6)	C6—C1—S1—N2	-1.0 (3)

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>
C13—H13 \cdots N2	0.93	2.28	2.901 (5)	124
N1—H1 \cdots O1 ⁱ	0.86	2.24	3.084 (4)	165
C5—H5 \cdots O1 ⁱ	0.93	2.51	3.396 (4)	159
C2—H2 \cdots O2 ⁱⁱ	0.93	2.42	3.302 (4)	158

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+3, -y+1, -z+1$.