

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## 2-Chloro-N-[(2-methylphenyl)sulfonyl]-acetamide

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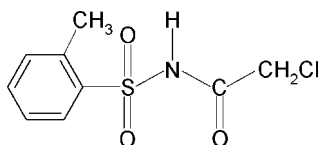
Received 18 January 2011; accepted 28 January 2011

 Key indicators: single-crystal X-ray study;  $T = 299$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.050;  $wR$  factor = 0.137; data-to-parameter ratio = 13.4.

In the title compound,  $\text{C}_9\text{H}_{10}\text{ClNO}_3\text{S}$ , the amide H atom is *syn* with respect to the *ortho*-methyl group in the benzene ring and the C–S–N–C torsion angle is  $-66.9(2)^\circ$ . An intramolecular N–H $\cdots$ Cl hydrogen bond occurs. The crystal structure features inversion-related dimers linked by pairs of N–H $\cdots$ O hydrogen bonds.

### Related literature

For the sulfanilamide moiety in sulfonamide drugs, see; Maren (1976). For its ability to form hydrogen bonds in the solid state, see; Yang & Guillory (1972). For hydrogen-bonding preferences of sulfonamides, see; Adsmund & Grant (2001). For the effect of substituents on the crystal structures of sulfonamides, see; Gowda *et al.* (2008*a,b*, 2010)



### Experimental

#### Crystal data

 $\text{C}_9\text{H}_{10}\text{ClNO}_3\text{S}$ 
 $M_r = 247.69$ 

 Triclinic,  $P\bar{1}$ 
 $a = 7.4439(8)$  Å

 $b = 7.5195(8)$  Å

 $c = 10.519(1)$  Å

 $\alpha = 93.64(1)^\circ$ 
 $\beta = 109.72(1)^\circ$ 
 $\gamma = 102.52(1)^\circ$ 
 $V = 535.07(10)$  Å<sup>3</sup>
 $Z = 2$ 

 Cu  $K\alpha$  radiation

 $\mu = 4.90$  mm<sup>-1</sup>
 $T = 299$  K

 $0.50 \times 0.40 \times 0.18$  mm

#### Data collection

Enraf–Nonius CAD-4

diffractometer

 Absorption correction:  $\psi$  scan

 (North *et al.*, 1968)

 $T_{\min} = 0.193$ ,  $T_{\max} = 0.473$ 

3727 measured reflections

1891 independent reflections

 1771 reflections with  $I > 2\sigma(I)$ 
 $R_{\text{int}} = 0.051$ 

3 standard reflections every 120 min

intensity decay: 0.5%

#### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.050$ 
 $wR(F^2) = 0.137$ 
 $S = 1.08$ 

1891 reflections

141 parameters

1 restraint

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.52$  e Å<sup>-3</sup>
 $\Delta\rho_{\text{min}} = -0.57$  e Å<sup>-3</sup>
**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^{\text{i}}$	0.79 (2)	2.32 (2)	3.087 (3)	166 (3)
$\text{N1}-\text{H1N}\cdots\text{Cl1}$	0.79 (2)	2.62 (3)	2.978 (2)	110 (2)

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

Data collection: *CAD-4-PC* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC*; data reduction: *REDU4* (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

KS thanks the University Grants Commission, Government of India, New Delhi, for the award of a research fellowship under its faculty improvement program.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DS2090).

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## supporting information

*Acta Cryst.* (2011). E67, o549 [doi:10.1107/S1600536811003655]

## 2-Chloro-*N*-[(2-methylphenyl)sulfonyl]acetamide

K. Shakuntala, Sabine Foro and B. Thimme Gowda

### S1. Comment

The molecular structures of sulfonamide drugs contain the sulfanilamide moiety (Maren, 1976). The propensity for hydrogen bonding in the solid state, due to the presence of various hydrogen bond donors and acceptors can give rise to polymorphism (Yang & Guillory, 1972). The hydrogen bonding preferences of sulfonamides has also been investigated (Adsmund & Grant, 2001). The nature and position of substituents play a significant role on the crystal structures of *N*-(aryl)sulfonamides (Gowda *et al.*, 2008*a,b*, 2010). As a part of studying the substituent effects on the structures of this class of compounds, the structure of 2-chloro-*N*-(2-methylphenylsulfonyl)-acetamide (I) has been determined. The conformations of the N—H and C=O bonds of the SO<sub>2</sub>—NH—CO—C segment in the structure are anti to each other (Fig. 1), similar to that observed in *N*-(phenylsulfonyl)acetamide (II)(Gowda *et al.*, 2010), *N*-(phenylsulfonyl)-2,2-dichloroacetamide (III) (Gowda *et al.*, 2008*b*) and *N*-(4-methylphenylsulfonyl)-2,2-dichloroacetamide (IV) (Gowda *et al.*, 2008*a*).

The molecule in (I) is bent at the *S*-atom with a C1—S1—N1—C7 torsion angle of -67.0 (3)°, compared to the values of -58.8 (4)° in (II), -66.3 (3)° in (III) and -71.1 (2)° in (IV). Further, the dihedral angle between the benzene ring and the SO<sub>2</sub>—NH—CO—C group in (I) is 78.9 (1)°, compared to the values of 89.0 (2)° in (II), 79.8 (1)° in (III) and 81.0 (1)° in (IV),

The structure exhibits both the intramolecular N—H···Cl and the intermolecular N—H···O(S) hydrogen bonds.

In the crystal structure, the pairs of intermolecular N—H···O hydrogen bonds (Table 1) link the molecules through inversion-related dimers into zigzag chains running in the *bc*-plane. Part of the crystal structure is shown in Fig. 2.

### S2. Experimental

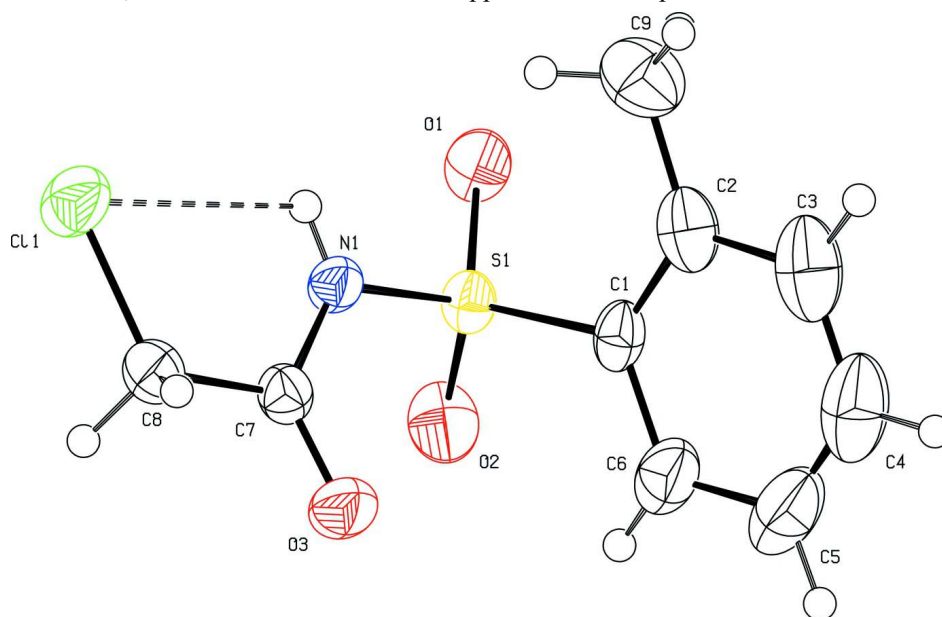
The title compound was prepared by refluxing 2-methylbenzenesulfonamide (0.10 mole) with an excess of chloroacetyl chloride (0.20 mole) for about an hour on a water bath. The reaction mixture was cooled and poured into ice cold water. The resulting solid was separated, washed thoroughly with water and dissolved in warm dilute sodium hydrogen carbonate solution. The title compound was reprecipitated by acidifying the filtered solution with glacial acetic acid. It was filtered, dried and recrystallized from ethanol. The purity of the compound was checked by determining its melting point. It was further characterized by recording its infrared spectra.

Prism like colorless single crystals of the title compound used in X-ray diffraction studies were obtained from a slow evaporation of an ethanolic solution of the compound.

### S3. Refinement

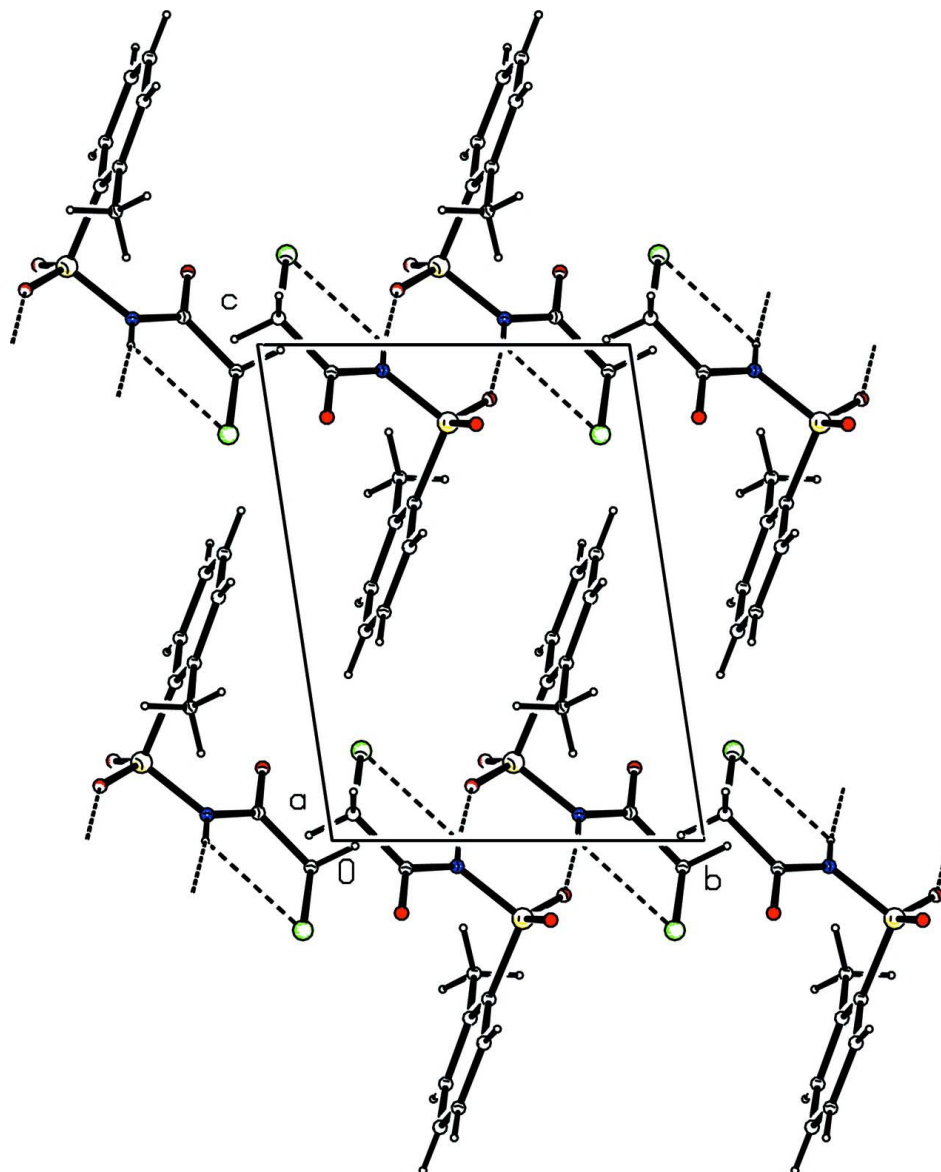
The H atom of the NH group was located in a difference map and later restrained to the distance N—H = 0.86 (3) Å. The other H atoms were positioned with idealized geometry using a riding model with C—H = 0.93–0.97 Å. All H atoms were refined with isotropic displacement parameters (set to 1.2 times of the  $U_{eq}$  of the parent atom).

The  $U^{ij}$  components of C3, C4 and C5 were restrained to approximate isotropic behavior.



**Figure 1**

Molecular structure of the title compound, showing the atom- labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

**Figure 2**

Molecular packing in the title compound. Hydrogen bonds are shown as dashed lines.

### 2-Chloro-*N*-[(2-methylphenyl)sulfonyl]acetamide

#### Crystal data

$C_9H_{10}ClNO_3S$

$M_r = 247.69$

Triclinic,  $P\bar{1}$

Hall symbol:  $-P\ 1$

$a = 7.4439$  (8) Å

$b = 7.5195$  (8) Å

$c = 10.519$  (1) Å

$\alpha = 93.64$  (1)°

$\beta = 109.72$  (1)°

$\gamma = 102.52$  (1)°

$V = 535.07$  (10) Å<sup>3</sup>

$Z = 2$

$F(000) = 256$

$D_x = 1.537$  Mg m<sup>-3</sup>

Cu  $K\alpha$  radiation,  $\lambda = 1.54180$  Å

Cell parameters from 25 reflections

$\theta = 7.0$ – $23.1$ °

$\mu = 4.90$  mm<sup>-1</sup>

$T = 299$  K

Prism, colourless

$0.50 \times 0.40 \times 0.18$  mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer	1891 independent reflections 1771 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.051$
Graphite monochromator	$\theta_{\text{max}} = 66.9^\circ$ , $\theta_{\text{min}} = 4.5^\circ$
$\omega/2\theta$ scans	$h = -8 \rightarrow 8$
Absorption correction: $\psi$ scan (North <i>et al.</i> , 1968)	$k = -8 \rightarrow 8$
$T_{\text{min}} = 0.193$ , $T_{\text{max}} = 0.473$	$l = -12 \rightarrow 12$
3727 measured reflections	3 standard reflections every 120 min intensity decay: 0.5%

*Refinement*

Refinement on $F^2$	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.050$	$w = 1/[\sigma^2(F_o^2) + (0.0853P)^2 + 0.2427P]$
$wR(F^2) = 0.137$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{\text{max}} < 0.001$
1891 reflections	$\Delta\rho_{\text{max}} = 0.52 \text{ e } \text{\AA}^{-3}$
141 parameters	$\Delta\rho_{\text{min}} = -0.57 \text{ e } \text{\AA}^{-3}$
1 restraint	Extinction correction: <i>SHELXL97</i> (Sheldrick, 2008), $F_c^* = kFc[1 + 0.001x \text{Fc}^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.028 (3)
Secondary atom site location: difference Fourier map	

*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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.4813 (4)	0.6426 (3)	0.3198 (2)	0.0356 (6)
C2	0.6844 (5)	0.7012 (3)	0.3580 (3)	0.0465 (7)
C3	0.7891 (6)	0.7927 (4)	0.4905 (3)	0.0674 (10)
H3	0.9259	0.8321	0.5206	0.081*
C4	0.6944 (8)	0.8259 (5)	0.5778 (3)	0.0764 (13)
H4	0.7682	0.8892	0.6653	0.092*
C5	0.4938 (8)	0.7680 (5)	0.5388 (3)	0.0754 (12)
H5	0.4320	0.7916	0.5992	0.090*
C6	0.3832 (6)	0.6737 (4)	0.4081 (3)	0.0531 (8)
H6	0.2468	0.6321	0.3800	0.064*
C7	0.2227 (4)	0.8123 (3)	0.0563 (2)	0.0336 (5)
C8	0.1973 (4)	0.9327 (3)	-0.0539 (3)	0.0397 (6)
H8A	0.2593	1.0596	-0.0110	0.048*
H8B	0.0574	0.9224	-0.0984	0.048*

C9	0.7974 (5)	0.6716 (5)	0.2685 (4)	0.0622 (8)
H9A	0.7241	0.6847	0.1766	0.075*
H9B	0.8181	0.5500	0.2704	0.075*
H9C	0.9226	0.7610	0.3010	0.075*
N1	0.3146 (3)	0.6745 (3)	0.05184 (19)	0.0327 (5)
H1N	0.366 (4)	0.660 (4)	-0.001 (3)	0.039*
O1	0.4251 (3)	0.3986 (2)	0.11017 (18)	0.0450 (5)
O2	0.1386 (3)	0.4445 (3)	0.1603 (2)	0.0516 (5)
O3	0.1615 (3)	0.8425 (3)	0.14615 (19)	0.0504 (5)
Cl1	0.29367 (12)	0.88507 (9)	-0.18091 (7)	0.0525 (3)
S1	0.32891 (8)	0.51904 (7)	0.15752 (5)	0.0320 (3)

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0487 (16)	0.0278 (10)	0.0267 (11)	0.0120 (10)	0.0075 (10)	0.0051 (8)
C2	0.0512 (18)	0.0318 (12)	0.0458 (14)	0.0066 (11)	0.0055 (12)	0.0113 (10)
C3	0.076 (3)	0.0459 (16)	0.0511 (18)	0.0051 (15)	-0.0078 (16)	0.0068 (13)
C4	0.112 (4)	0.0511 (18)	0.0400 (17)	0.017 (2)	-0.0020 (19)	-0.0004 (13)
C5	0.137 (4)	0.070 (2)	0.0381 (16)	0.051 (2)	0.039 (2)	0.0132 (14)
C6	0.076 (2)	0.0538 (16)	0.0392 (14)	0.0305 (15)	0.0231 (14)	0.0143 (11)
C7	0.0357 (13)	0.0325 (11)	0.0310 (11)	0.0118 (9)	0.0084 (9)	0.0030 (8)
C8	0.0451 (16)	0.0363 (12)	0.0417 (13)	0.0181 (11)	0.0150 (11)	0.0101 (10)
C9	0.0466 (19)	0.0606 (18)	0.076 (2)	0.0087 (14)	0.0209 (15)	0.0130 (15)
N1	0.0407 (12)	0.0335 (10)	0.0287 (9)	0.0161 (8)	0.0141 (8)	0.0063 (8)
O1	0.0661 (14)	0.0323 (9)	0.0412 (9)	0.0231 (9)	0.0185 (9)	0.0057 (7)
O2	0.0432 (12)	0.0498 (11)	0.0537 (11)	-0.0001 (9)	0.0134 (9)	0.0151 (8)
O3	0.0648 (14)	0.0597 (12)	0.0438 (10)	0.0355 (10)	0.0277 (10)	0.0130 (8)
Cl1	0.0744 (6)	0.0547 (5)	0.0462 (4)	0.0325 (4)	0.0314 (4)	0.0216 (3)
S1	0.0384 (4)	0.0259 (4)	0.0298 (4)	0.0087 (3)	0.0093 (3)	0.0049 (2)

*Geometric parameters (Å, °)*

C1—C2	1.386 (4)	C7—N1	1.367 (3)
C1—C6	1.398 (4)	C7—C8	1.502 (3)
C1—S1	1.760 (2)	C8—Cl1	1.768 (3)
C2—C3	1.392 (4)	C8—H8A	0.9700
C2—C9	1.494 (5)	C8—H8B	0.9700
C3—C4	1.374 (7)	C9—H9A	0.9600
C3—H3	0.9300	C9—H9B	0.9600
C4—C5	1.367 (6)	C9—H9C	0.9600
C4—H4	0.9300	N1—S1	1.6590 (19)
C5—C6	1.389 (5)	N1—H1N	0.79 (2)
C5—H5	0.9300	O1—S1	1.4303 (18)
C6—H6	0.9300	O2—S1	1.417 (2)
C7—O3	1.208 (3)		
C2—C1—C6	122.5 (2)	C7—C8—Cl1	116.35 (17)

C2—C1—S1	122.3 (2)	C7—C8—H8A	108.2
C6—C1—S1	115.3 (2)	C11—C8—H8A	108.2
C1—C2—C3	116.8 (3)	C7—C8—H8B	108.2
C1—C2—C9	124.9 (2)	C11—C8—H8B	108.2
C3—C2—C9	118.3 (3)	H8A—C8—H8B	107.4
C4—C3—C2	121.3 (4)	C2—C9—H9A	109.5
C4—C3—H3	119.4	C2—C9—H9B	109.5
C2—C3—H3	119.4	H9A—C9—H9B	109.5
C5—C4—C3	121.4 (3)	C2—C9—H9C	109.5
C5—C4—H4	119.3	H9A—C9—H9C	109.5
C3—C4—H4	119.3	H9B—C9—H9C	109.5
C4—C5—C6	119.4 (3)	C7—N1—S1	123.27 (17)
C4—C5—H5	120.3	C7—N1—H1N	124 (2)
C6—C5—H5	120.3	S1—N1—H1N	113 (2)
C5—C6—C1	118.7 (4)	O2—S1—O1	118.71 (12)
C5—C6—H6	120.7	O2—S1—N1	108.92 (11)
C1—C6—H6	120.7	O1—S1—N1	103.64 (10)
O3—C7—N1	122.6 (2)	O2—S1—C1	108.50 (12)
O3—C7—C8	118.3 (2)	O1—S1—C1	110.83 (12)
N1—C7—C8	119.1 (2)	N1—S1—C1	105.34 (10)
C6—C1—C2—C3	0.5 (4)	N1—C7—C8—C11	-0.7 (3)
S1—C1—C2—C3	-178.1 (2)	O3—C7—N1—S1	7.4 (4)
C6—C1—C2—C9	179.8 (3)	C8—C7—N1—S1	-172.97 (18)
S1—C1—C2—C9	1.2 (4)	C7—N1—S1—O2	49.3 (2)
C1—C2—C3—C4	-1.2 (4)	C7—N1—S1—O1	176.60 (19)
C9—C2—C3—C4	179.4 (3)	C7—N1—S1—C1	-66.9 (2)
C2—C3—C4—C5	1.0 (5)	C2—C1—S1—O2	167.1 (2)
C3—C4—C5—C6	0.0 (5)	C6—C1—S1—O2	-11.5 (2)
C4—C5—C6—C1	-0.7 (5)	C2—C1—S1—O1	35.1 (2)
C2—C1—C6—C5	0.4 (4)	C6—C1—S1—O1	-143.56 (19)
S1—C1—C6—C5	179.1 (2)	C2—C1—S1—N1	-76.3 (2)
O3—C7—C8—C11	178.8 (2)	C6—C1—S1—N1	105.0 (2)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1N...O1 <sup>i</sup>	0.79 (2)	2.32 (2)	3.087 (3)	166 (3)
N1—H1N...C11	0.79 (2)	2.62 (3)	2.978 (2)	110 (2)

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