

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

N-(2,4-Dichlorophenyl)-4-methylbenzenesulfonamide

K. Shakuntala,^a Sabine Foro^b and B. Thimme Gowda^{a*}

^aDepartment of Chemistry, Mangalore University, Mangalagangothri 574 199, Mangalore, India, and ^bInstitute of Materials Science, Darmstadt University of Technology, Petersenstrasse 23, D-64287 Darmstadt, Germany

Correspondence e-mail: gowdabt@yahoo.com

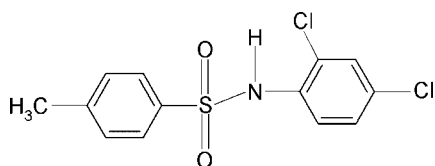
Received 24 July 2011; accepted 1 August 2011

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.036; wR factor = 0.103; data-to-parameter ratio = 16.5.

The molecule of the title compound, $\text{C}_{13}\text{H}_{11}\text{Cl}_2\text{NO}_2\text{S}$, is bent at the S atom with a $\text{C}-\text{SO}_2-\text{NH}-\text{C}$ torsion angle of $-69.07(16)^\circ$. The sulfonyl and aniline rings are rotated relative to each other by $53.0(1)^\circ$. In the crystal, pairs of $\text{N}-\text{H}\cdots\text{O}(\text{S})$ hydrogen bonds link the molecules into centrosymmetric dimers.

Related literature

For hydrogen-bonding modes of sulfonamides, see: Admond & Grant (2001). For studies on the effects of substituents on the structures and other aspects of *N*-(aryl)-amides, see: Arjunan *et al.* (2004); Gowda *et al.* (2000, 2006), on *N*-(aryl)-methanesulfonamides, see: Gowda *et al.* (2007) and on *N*-(aryl)-arylsulfonamides, see: Gelbrich *et al.* (2007); Perlovich *et al.* (2006); Shakuntala *et al.* (2010). For the preparation of the title compound, see: Shetty & Gowda (2005)



Experimental

Crystal data

$\text{C}_{13}\text{H}_{11}\text{Cl}_2\text{NO}_2\text{S}$
 $M_r = 316.19$
 Monoclinic, $P2_1/c$
 $a = 11.385(1)$ Å
 $b = 11.959(1)$ Å
 $c = 11.428(1)$ Å
 $\beta = 112.87(2)^\circ$

$V = 1433.6(2)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.59$ mm⁻¹
 $T = 293$ K
 $0.44 \times 0.38 \times 0.28$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
 Absorption correction: multi-scan (*CrysAlis RED*; Oxford)

Diffraction, 2009)
 $T_{\min} = 0.780$, $T_{\max} = 0.851$
 5752 measured reflections
 2924 independent reflections
 2361 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.013$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.103$
 $S = 1.04$
 2924 reflections
 177 parameters
 1 restraint

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.28$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.38$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^i$	0.85 (2)	2.21 (2)	3.024 (2)	160 (2)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; 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: BT5590).

References

- Admond, D. A. & Grant, D. J. W. (2001). *J. Pharm. Sci.* **90**, 2058–2077.
 Arjunan, V., Mohan, S., Subramanian, S. & Gowda, B. T. (2004). *Spectrochim. Acta Part A*, **60**, 1141–1159.
 Gelbrich, T., Hursthouse, M. B. & Threlfall, T. L. (2007). *Acta Cryst.* **B63**, 621–632.
 Gowda, B. T., Foro, S. & Fuess, H. (2007). *Acta Cryst.* **E63**, o2339.
 Gowda, B. T., Kozisek, J. & Fuess, H. (2006). *Z. Naturforsch. Teil A*, **55**, 588–594.
 Gowda, B. T., Kumar, B. H. A. & Fuess, H. (2000). *Z. Naturforsch. Teil A*, **55**, 721–728.
 Oxford Diffraction (2009). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Yarnton, England.
 Perlovich, G. L., Tkachev, V. V., Schaper, K.-J. & Raevsky, O. A. (2006). *Acta Cryst.* **E62**, o780–o782.
 Shakuntala, K., Foro, S., Gowda, B. T., Nirmala, P. G. & Fuess, H. (2010). *Acta Cryst.* **E66**, o3062.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Shetty, M. & Gowda, B. T. (2005). *Z. Naturforsch. Teil A*, **59**, 113–120.
 Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

supporting information

Acta Cryst. (2011). E67, o2252 [doi:10.1107/S1600536811030819]

N-(2,4-Dichlorophenyl)-4-methylbenzenesulfonamide

K. Shakuntala, Sabine Foro and B. Thimme Gowda

S1. Comment

The amide and sulfonamide moieties are the constituents of many biologically significant compounds. The hydrogen bonding preferences of sulfonamides have been investigated (Adsmund & Grant, 2001). As part of our work on the substituent effects on the structures and other aspects of *N*-(aryl)-amides (Arjunan *et al.*, 2004; Gowda *et al.*, 2000, 2006), *N*-(aryl)-methanesulfonamides (Gowda *et al.*, 2007) and *N*-(aryl)-arylsulfonamides (Shakuntala *et al.*, 2010), in the present work, the crystal structure of *N*-(2,4-dichlorophenyl)-4-methylbenzenesulfonamide (I) has been determined (Fig. 1).

The conformation of the N—H bond in the C—SO₂—NH—C segment and the *ortho*-chloro group in the anilino benzene ring are *syn* to each other. The molecule is bent at the S atom with the C—SO₂—NH—C torsion angle of -69.1 (2)°, compared to the values of 65.4 (2) and -61.7 (2)° in the two molecules of *N*-(2,3-dichlorophenyl)-4-methylbenzenesulfonamide (II) (Shakuntala *et al.*, 2010).

The sulfonyl and the aniline benzene rings in (I) are tilted relative to each other by 53.0 (1)°, compared to the values of 76.0 (1) and 79.9 (1)° in the two molecules of (II). The other bond parameters in (I) are similar to those observed in (II) and other aryl sulfonamides (Perlovich *et al.*, 2006; Gelbrich *et al.*, 2007).

In the crystal, the N—H···O hydrogen bonds (Table 1) pack the molecules into zigzag chains in the direction parallel to *b*-axis (Fig. 2).

S2. Experimental

The solution of toluene (10 ml) in chloroform (40 ml) was treated dropwise with chlorosulfonic acid (25 ml) at 0 °C. After the initial evolution of hydrogen chloride subsided, the reaction mixture was brought to room temperature and poured into crushed ice in a beaker. The chloroform layer was separated, washed with cold water and allowed to evaporate slowly. The residual 4-methylbenzenesulfonylchloride was treated with 2,4-dichloroaniline in the stoichiometric ratio and boiled for ten minutes. The reaction mixture was then cooled to room temperature and added to ice cold water (100 ml). The resultant *N*-(2,4-dichlorophenyl)-4-methylbenzenesulfonamide was filtered under suction and washed thoroughly with cold water. It was then recrystallized to constant melting point from dilute ethanol. The purity of the compound was checked and characterized by recording its infrared and NMR spectra (Shetty & Gowda, 2005).

The prism like colourless single crystals used in X-ray diffraction studies were grown in ethanolic solution by a slow evaporation at room temperature.

S3. Refinement

The H atom of the NH group was located in a difference map and later restrained to N—H = 0.86 (2) %A. The other H atoms were positioned with idealized geometry using a riding model with the aromatic C—H = 0.93 Å and methyl C—H

= 0.96 Å. The $U_{\text{iso}}(\text{H})$ values were set at $1.2U_{\text{eq}}(\text{C-aromatic, N})$ and $1.5U_{\text{eq}}(\text{C-methyl})$.

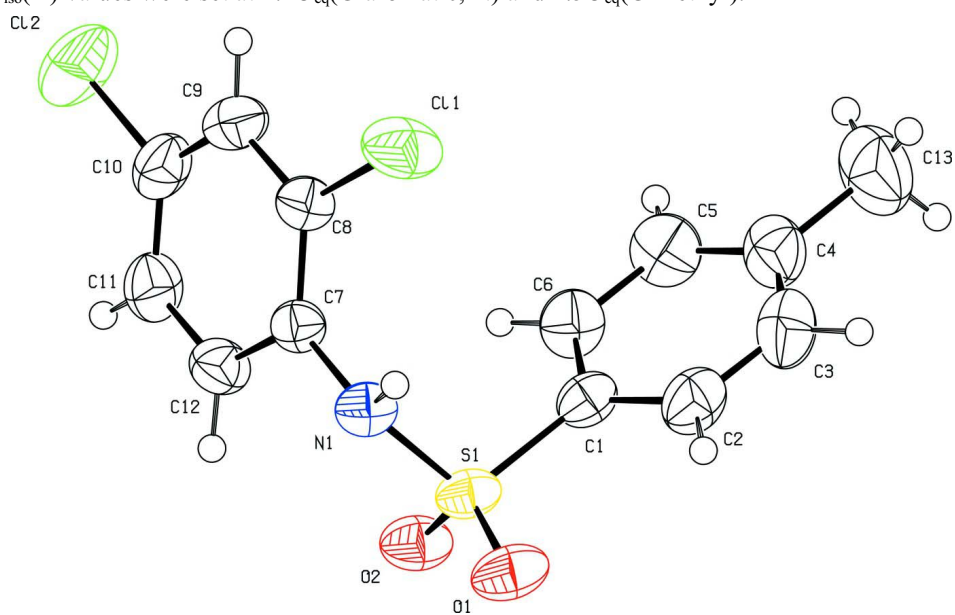


Figure 1

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

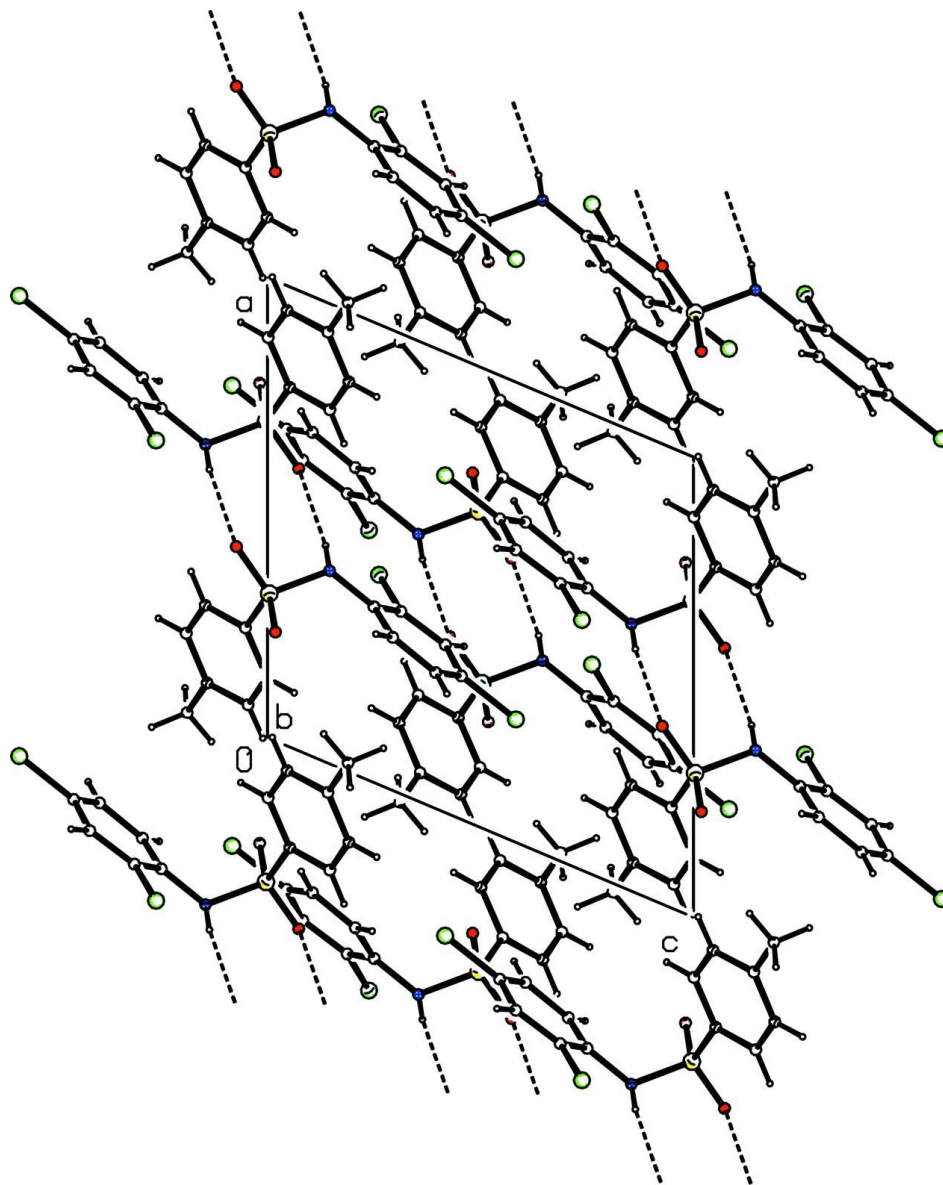


Figure 2

Molecular packing of (I) with hydrogen bonding shown as dashed lines.

***N*-(2,4-Dichlorophenyl)-4-methylbenzenesulfonamide**

Crystal data

$C_{13}H_{11}Cl_2NO_2S$

$M_r = 316.19$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 11.385\ (1)\ \text{\AA}$

$b = 11.959\ (1)\ \text{\AA}$

$c = 11.428\ (1)\ \text{\AA}$

$\beta = 112.87\ (2)^\circ$

$V = 1433.6\ (2)\ \text{\AA}^3$

$Z = 4$

$F(000) = 648$

$D_x = 1.465\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2440 reflections

$\theta = 2.6\text{--}27.7^\circ$

$\mu = 0.59\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Prism, colourless

$0.44 \times 0.38 \times 0.28\ \text{mm}$

Data collection

Oxford Diffraction Xcalibur
 diffractometer with a Sapphire CCD detector
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 Rotation method data acquisition using ω and ϕ
 scans
 Absorption correction: multi-scan
 (*CrysAlis RED*; Oxford Diffraction, 2009)
 $T_{\min} = 0.780$, $T_{\max} = 0.851$

5752 measured reflections
 2924 independent reflections
 2361 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.013$
 $\theta_{\max} = 26.4^\circ$, $\theta_{\min} = 2.6^\circ$
 $h = -14 \rightarrow 14$
 $k = -10 \rightarrow 14$
 $l = -14 \rightarrow 11$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.103$
 $S = 1.04$
 2924 reflections
 177 parameters
 1 restraint
 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
 $w = 1/[\sigma^2(F_o^2) + (0.0569P)^2 + 0.3942P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.018$
 $\Delta\rho_{\max} = 0.28 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.38 \text{ e } \text{\AA}^{-3}$
 Extinction correction: *SHELXL97* (Sheldrick,
 2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.070 (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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.21863 (18)	-0.05135 (17)	-0.05214 (18)	0.0414 (4)
C2	0.2305 (2)	-0.1166 (2)	-0.1463 (2)	0.0564 (6)
H2	0.2911	-0.0991	-0.1794	0.068*
C3	0.1525 (2)	-0.2075 (2)	-0.1910 (2)	0.0660 (6)
H3	0.1595	-0.2500	-0.2561	0.079*
C4	0.0635 (2)	-0.2378 (2)	-0.1417 (2)	0.0607 (6)
C5	0.0538 (2)	-0.1719 (2)	-0.0458 (2)	0.0617 (6)
H5	-0.0051	-0.1907	-0.0111	0.074*
C6	0.1300 (2)	-0.07897 (19)	-0.0012 (2)	0.0525 (5)
H6	0.1220	-0.0352	0.0626	0.063*
C7	0.38204 (17)	0.02375 (15)	0.24980 (17)	0.0369 (4)
C8	0.39658 (18)	-0.07796 (15)	0.31288 (18)	0.0418 (4)
C9	0.3646 (2)	-0.09025 (18)	0.4174 (2)	0.0502 (5)
H9	0.3785	-0.1577	0.4612	0.060*

C10	0.3118 (2)	-0.0006 (2)	0.45504 (19)	0.0522 (5)
C11	0.2956 (2)	0.10096 (19)	0.3946 (2)	0.0536 (5)
H11	0.2600	0.1608	0.4212	0.064*
C12	0.3327 (2)	0.11299 (17)	0.29395 (19)	0.0469 (5)
H12	0.3245	0.1824	0.2548	0.056*
C13	-0.0193 (3)	-0.3395 (3)	-0.1891 (3)	0.0923 (10)
H13A	0.0322	-0.4014	-0.1944	0.111*
H13B	-0.0835	-0.3245	-0.2717	0.111*
H13C	-0.0593	-0.3577	-0.1315	0.111*
N1	0.41946 (15)	0.03784 (14)	0.14553 (15)	0.0404 (4)
H1N	0.4704 (18)	-0.0102 (15)	0.136 (2)	0.048*
O1	0.38666 (14)	0.08540 (13)	-0.07316 (14)	0.0563 (4)
O2	0.23685 (14)	0.15510 (12)	0.01841 (14)	0.0510 (4)
Cl1	0.45623 (6)	-0.19221 (5)	0.26176 (6)	0.0665 (2)
Cl2	0.26736 (7)	-0.01719 (8)	0.58308 (6)	0.0843 (3)
S1	0.31493 (4)	0.06739 (4)	0.00383 (5)	0.04119 (17)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0392 (9)	0.0493 (11)	0.0380 (9)	0.0077 (8)	0.0176 (8)	0.0066 (8)
C2	0.0641 (13)	0.0662 (14)	0.0471 (11)	0.0042 (12)	0.0307 (11)	-0.0004 (11)
C3	0.0703 (16)	0.0719 (16)	0.0534 (13)	0.0024 (13)	0.0213 (12)	-0.0156 (12)
C4	0.0484 (12)	0.0623 (14)	0.0561 (13)	0.0007 (11)	0.0035 (10)	-0.0041 (11)
C5	0.0430 (11)	0.0757 (16)	0.0679 (14)	-0.0083 (11)	0.0231 (11)	-0.0012 (13)
C6	0.0468 (11)	0.0630 (14)	0.0561 (12)	-0.0011 (10)	0.0291 (10)	-0.0048 (10)
C7	0.0346 (9)	0.0384 (9)	0.0394 (9)	-0.0021 (8)	0.0162 (8)	0.0008 (8)
C8	0.0428 (10)	0.0384 (10)	0.0455 (10)	0.0012 (8)	0.0188 (8)	0.0020 (8)
C9	0.0547 (12)	0.0511 (12)	0.0464 (11)	-0.0006 (10)	0.0215 (10)	0.0104 (9)
C10	0.0545 (12)	0.0675 (14)	0.0395 (10)	-0.0056 (11)	0.0239 (9)	-0.0030 (10)
C11	0.0616 (13)	0.0539 (12)	0.0516 (12)	0.0034 (11)	0.0287 (11)	-0.0109 (10)
C12	0.0559 (12)	0.0376 (10)	0.0518 (11)	0.0019 (9)	0.0259 (10)	0.0000 (9)
C13	0.0752 (19)	0.086 (2)	0.095 (2)	-0.0204 (17)	0.0106 (16)	-0.0174 (17)
N1	0.0374 (8)	0.0428 (9)	0.0468 (9)	0.0039 (7)	0.0227 (7)	0.0048 (7)
O1	0.0604 (9)	0.0663 (10)	0.0574 (9)	0.0063 (8)	0.0395 (8)	0.0187 (7)
O2	0.0544 (8)	0.0441 (8)	0.0608 (9)	0.0134 (7)	0.0294 (7)	0.0134 (7)
Cl1	0.0877 (5)	0.0423 (3)	0.0857 (4)	0.0176 (3)	0.0513 (4)	0.0088 (3)
Cl2	0.0955 (5)	0.1192 (6)	0.0568 (4)	-0.0017 (5)	0.0500 (4)	0.0039 (4)
S1	0.0430 (3)	0.0440 (3)	0.0447 (3)	0.0062 (2)	0.0259 (2)	0.0116 (2)

Geometric parameters (Å, °)

C1—C2	1.378 (3)	C8—C11	1.7256 (19)
C1—C6	1.387 (3)	C9—C10	1.377 (3)
C1—S1	1.756 (2)	C9—H9	0.9300
C2—C3	1.371 (3)	C10—C11	1.374 (3)
C2—H2	0.9300	C10—Cl2	1.736 (2)
C3—C4	1.385 (3)	C11—C12	1.378 (3)

C3—H3	0.9300	C11—H11	0.9300
C4—C5	1.389 (3)	C12—H12	0.9300
C4—C13	1.505 (4)	C13—H13A	0.9600
C5—C6	1.380 (3)	C13—H13B	0.9600
C5—H5	0.9300	C13—H13C	0.9600
C6—H6	0.9300	N1—S1	1.6326 (17)
C7—C12	1.389 (3)	N1—H1N	0.852 (15)
C7—C8	1.391 (3)	O1—S1	1.4304 (13)
C7—N1	1.423 (2)	O2—S1	1.4260 (14)
C8—C9	1.385 (3)		
C2—C1—C6	120.1 (2)	C8—C9—H9	120.7
C2—C1—S1	120.38 (16)	C11—C10—C9	121.39 (19)
C6—C1—S1	119.53 (16)	C11—C10—C12	119.92 (17)
C3—C2—C1	119.6 (2)	C9—C10—C12	118.68 (17)
C3—C2—H2	120.2	C10—C11—C12	119.14 (19)
C1—C2—H2	120.2	C10—C11—H11	120.4
C2—C3—C4	121.8 (2)	C12—C11—H11	120.4
C2—C3—H3	119.1	C11—C12—C7	121.42 (19)
C4—C3—H3	119.1	C11—C12—H12	119.3
C3—C4—C5	117.9 (2)	C7—C12—H12	119.3
C3—C4—C13	121.3 (2)	C4—C13—H13A	109.5
C5—C4—C13	120.8 (2)	C4—C13—H13B	109.5
C6—C5—C4	121.2 (2)	H13A—C13—H13B	109.5
C6—C5—H5	119.4	C4—C13—H13C	109.5
C4—C5—H5	119.4	H13A—C13—H13C	109.5
C5—C6—C1	119.5 (2)	H13B—C13—H13C	109.5
C5—C6—H6	120.3	C7—N1—S1	121.08 (12)
C1—C6—H6	120.3	C7—N1—H1N	117.6 (15)
C12—C7—C8	117.85 (17)	S1—N1—H1N	107.0 (15)
C12—C7—N1	120.55 (16)	O2—S1—O1	119.47 (9)
C8—C7—N1	121.58 (16)	O2—S1—N1	106.89 (9)
C9—C8—C7	121.44 (18)	O1—S1—N1	105.80 (9)
C9—C8—C11	118.54 (15)	O2—S1—C1	107.95 (9)
C7—C8—C11	120.02 (14)	O1—S1—C1	108.83 (9)
C10—C9—C8	118.67 (19)	N1—S1—C1	107.31 (9)
C10—C9—H9	120.7		
C6—C1—C2—C3	1.3 (3)	C8—C9—C10—C12	178.11 (16)
S1—C1—C2—C3	-178.49 (18)	C9—C10—C11—C12	0.1 (3)
C1—C2—C3—C4	-1.7 (4)	C12—C10—C11—C12	179.30 (17)
C2—C3—C4—C5	0.9 (4)	C10—C11—C12—C7	2.2 (3)
C2—C3—C4—C13	-178.4 (2)	C8—C7—C12—C11	-1.9 (3)
C3—C4—C5—C6	0.2 (4)	N1—C7—C12—C11	179.82 (18)
C13—C4—C5—C6	179.5 (2)	C12—C7—N1—S1	-65.1 (2)
C4—C5—C6—C1	-0.6 (3)	C8—C7—N1—S1	116.64 (18)
C2—C1—C6—C5	-0.2 (3)	C7—N1—S1—O2	46.53 (16)
S1—C1—C6—C5	179.58 (17)	C7—N1—S1—O1	174.86 (14)

C12—C7—C8—C9	-0.8 (3)	C7—N1—S1—C1	-69.07 (16)
N1—C7—C8—C9	177.50 (18)	C2—C1—S1—O2	137.68 (17)
C12—C7—C8—C11	179.49 (15)	C6—C1—S1—O2	-42.10 (18)
N1—C7—C8—C11	-2.2 (3)	C2—C1—S1—O1	6.6 (2)
C7—C8—C9—C10	3.0 (3)	C6—C1—S1—O1	-173.16 (16)
C11—C8—C9—C10	-177.23 (17)	C2—C1—S1—N1	-107.43 (17)
C8—C9—C10—C11	-2.7 (3)	C6—C1—S1—N1	72.78 (18)

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 ⁱ	0.85 (2)	2.21 (2)	3.024 (2)	160 (2)

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