

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

N-(2,6-Dimethylphenyl)-4-methylbenzenesulfonamide

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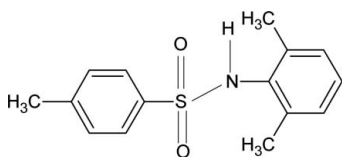
Received 8 April 2010; accepted 20 April 2010

Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.042; wR factor = 0.116; data-to-parameter ratio = 14.5.

In the title compound, $\text{C}_{15}\text{H}_{17}\text{NO}_2\text{S}$, the molecule is bent at the S atom, the $\text{C}-\text{SO}_2-\text{NH}-\text{C}$ torsion angle being $88.0(2)^\circ$. The dihedral angle between the two aromatic rings is $49.8(1)^\circ$. In the crystal, molecules are linked into zigzag chains parallel to the a axis via $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For the preparation of the title compound, see: Shetty & Gowda (2005). For our study of the effect of substituents on the structures of *N*-(aryl)-arylsulfonamides, see: Gowda *et al.* (2008, 2009, 2010). For related structures, see: Gelbrich *et al.* (2007); Perlovich *et al.* (2006).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{17}\text{NO}_2\text{S}$
 $M_r = 275.36$
Monoclinic, $P2_1/n$
 $a = 5.1412(5)$ Å

$b = 17.310(2)$ Å
 $c = 16.429(2)$ Å
 $\beta = 96.65(1)^\circ$
 $V = 1452.2(3)$ Å³

$Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.22$ mm⁻¹

$T = 299$ K
 $0.46 \times 0.32 \times 0.14$ mm

Data collection

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

Diffraction, 2009)
 $T_{\min} = 0.906$, $T_{\max} = 0.970$
7741 measured reflections
2580 independent reflections
2090 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.052$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.116$
 $S = 1.04$
2580 reflections
178 parameters
1 restraint

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.25$ e Å⁻³
 $\Delta\rho_{\min} = -0.36$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^i$	0.85 (1)	2.20 (1)	3.040 (2)	169 (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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: VM2025).

References

- Gelbrich, T., Hursthouse, M. B. & Threlfall, T. L. (2007). *Acta Cryst.* **B63**, 621–632.
Gowda, B. T., Foro, S., Babitha, K. S. & Fuess, H. (2008). *Acta Cryst.* **E64**, o1691.
Gowda, B. T., Foro, S., Nirmala, P. G. & Fuess, H. (2010). *Acta Cryst.* **E66**, o144.
Gowda, B. T., Foro, S., Nirmala, P. G., Teruo, H. & Fuess, H. (2009). *Acta Cryst.* **E65**, o1219.
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.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Shetty, M. & Gowda, B. T. (2005). *Z. Naturforsch. Teil A*, **60**, 113–120.
Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

supporting information

Acta Cryst. (2010). E66, o1168 [https://doi.org/10.1107/S1600536810014571]

***N*-(2,6-Dimethylphenyl)-4-methylbenzenesulfonamide**

P. G. Nirmala, B. Thimme Gowda, Sabine Foro and Hartmut Fuess

S1. Comment

In the present work, as part of a study of the effect of substituents on the crystal structures of *N*-(aryl)-arylsulfonamides (Gowda *et al.*, 2008, 2009, 2010), the structure of *N*-(2,6-dimethylphenyl)-4-methylbenzenesulfonamide (I) has been determined. The molecule is bent at the *S* atom (Fig. 1) with the C1—SO₂—NH—C7 torsion angle of 88.0 (2)°, compared to the values of -51.6 (3)° in *N*-(phenyl)-4-methylbenzenesulfonamide (II) (Gowda *et al.*, 2009), -78.7 (2)° in *N*-(2,6-dimethylphenyl)-benzenesulfonamide (III) (Gowda *et al.*, 2008) and -61.0 (2)° in *N*-(2,5-dimethylphenyl)-4-methylbenzenesulfonamide (IV), -61.8 (2)° in *N*-(3,4-dimethylphenyl)-4-methylbenzenesulfonamide (V) and 56.8 (2)° in *N*-(3,5-dimethylphenyl)-4-methylbenzenesulfonamide (VI) (Gowda *et al.*, 2010).

The two benzene rings in (I) are tilted relative to each other by 49.8 (1)°, compared to the values of 68.4 (1)° in (II), 44.9 (1)° in (III), 49.4 (1)° in (IV), 47.8 (1)° in (V) and 53.9 (1)° in (VI). The other bond parameters are similar to those observed in (II), (III), (IV), (V), (VI) and other aryl sulfonamides (Perlovich *et al.*, 2006; Gelbrich *et al.*, 2007).

In the crystal structure, the intermolecular N—H...O hydrogen bonds (Table 1) link the molecules into infinite zig-zag chains running parallel to the *a*-axis. Part of the crystal structure is shown in Fig. 2.

S2. Experimental

4-Methylbenzenesulfonylchloride was obtained by treating the solution of toluene (10 ml) in chloroform (40 ml) with chlorosulfonic acid (25 ml) by the procedure reported earlier (Gowda *et al.*, 2010). 4-Methylbenzenesulfonylchloride was then treated with 2,6-dimethylaniline in the stoichiometric ratio to obtain *N*-(2,6-dimethylphenyl)-4-methylbenzenesulfonamide. The latter was recrystallized to constant melting point (110 °C) 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 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 (1) Å. The other H atoms were positioned with idealized geometry using a riding model with C—H = 0.93–0.96 Å. All H atoms were refined with isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).

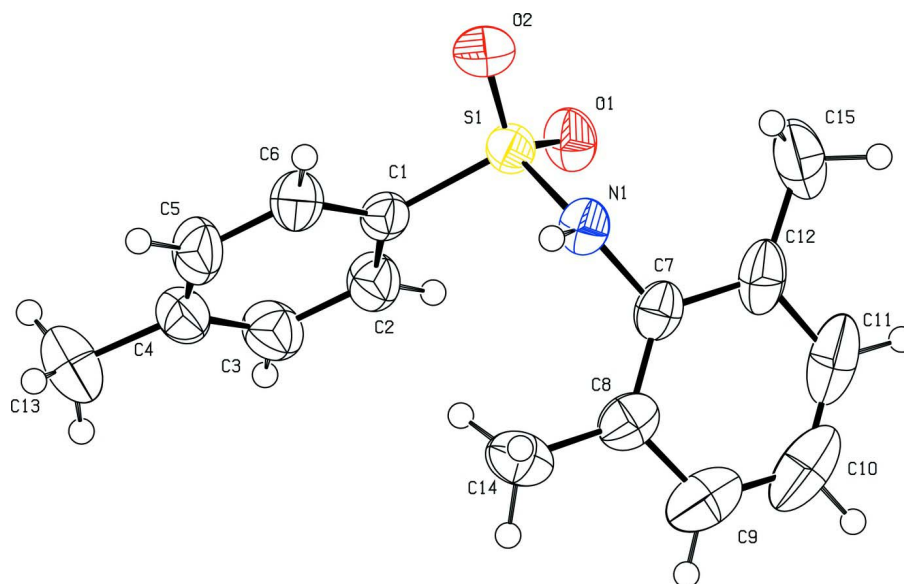


Figure 1

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

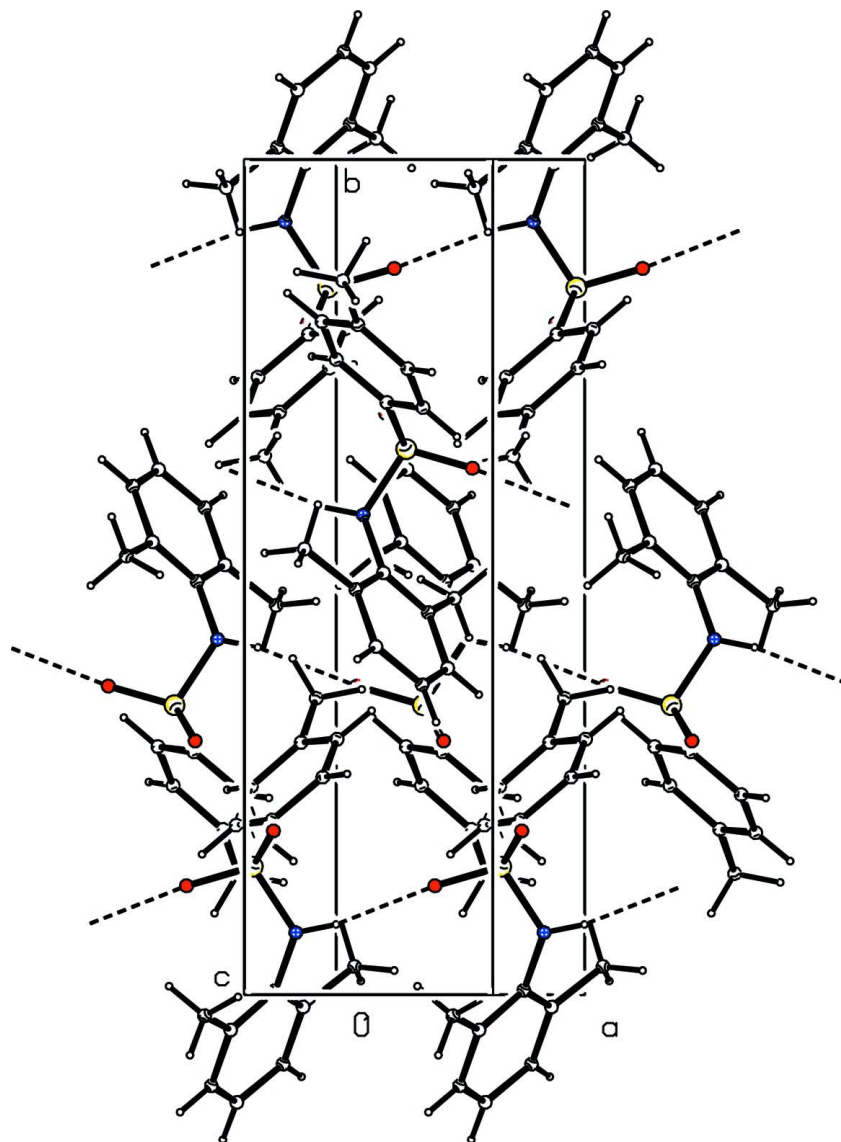


Figure 2
Molecular packing of (I) with hydrogen bonding shown as dashed lines.

N-(2,6-Dimethylphenyl)-4-methylbenzenesulfonamide

Crystal data

$C_{15}H_{17}NO_2S$

$M_r = 275.36$

Monoclinic, $P2_1/n$

Hall symbol: $-P 2_1n$

$a = 5.1412 (5) \text{ \AA}$

$b = 17.310 (2) \text{ \AA}$

$c = 16.429 (2) \text{ \AA}$

$\beta = 96.65 (1)^\circ$

$V = 1452.2 (3) \text{ \AA}^3$

$Z = 4$

$F(000) = 584$

$D_x = 1.259 \text{ Mg m}^{-3}$

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

Cell parameters from 2102 reflections

$\theta = 2.5\text{--}27.7^\circ$

$\mu = 0.22 \text{ mm}^{-1}$

$T = 299 \text{ K}$

Prism, colourless

$0.46 \times 0.32 \times 0.14 \text{ mm}$

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
phi scans.
Absorption correction: multi-scan
(*CrysAlis RED*; Oxford Diffraction, 2009)
 $T_{\min} = 0.906$, $T_{\max} = 0.970$

7741 measured reflections
2580 independent reflections
2090 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.052$
 $\theta_{\max} = 25.4^\circ$, $\theta_{\min} = 2.5^\circ$
 $h = -6 \rightarrow 6$
 $k = -20 \rightarrow 20$
 $l = -18 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.116$
 $S = 1.04$
2580 reflections
178 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
 $w = 1/[\sigma^2(F_o^2) + (0.0536P)^2 + 0.6128P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.011$
 $\Delta\rho_{\max} = 0.25 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.36 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. *CrysAlis RED* (Oxford Diffraction, 2009) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.0076 (4)	0.79081 (11)	0.32294 (13)	0.0373 (5)
C2	0.1791 (5)	0.79695 (14)	0.39383 (14)	0.0499 (6)
H2	0.3173	0.8319	0.3971	0.060*
C3	0.1426 (5)	0.75041 (15)	0.45988 (15)	0.0571 (6)
H3	0.2573	0.7546	0.5078	0.069*
C4	-0.0596 (5)	0.69793 (14)	0.45655 (15)	0.0537 (6)
C5	-0.2303 (5)	0.69408 (15)	0.38508 (17)	0.0580 (7)
H5	-0.3706	0.6599	0.3821	0.070*
C6	-0.1976 (4)	0.73955 (13)	0.31845 (15)	0.0487 (5)
H6	-0.3133	0.7357	0.2707	0.058*
C7	-0.0218 (4)	0.99486 (12)	0.27900 (14)	0.0416 (5)
C8	-0.1053 (4)	1.01478 (14)	0.35381 (15)	0.0499 (6)
C9	-0.0115 (6)	1.08314 (17)	0.39028 (19)	0.0747 (8)

H9	-0.0666	1.0982	0.4398	0.090*
C10	0.1617 (7)	1.12894 (18)	0.3544 (3)	0.0913 (11)
H10	0.2274	1.1736	0.3807	0.110*
C11	0.2372 (6)	1.10901 (16)	0.2804 (3)	0.0823 (10)
H11	0.3537	1.1407	0.2567	0.099*
C12	0.1440 (5)	1.04222 (13)	0.23938 (18)	0.0575 (7)
C13	-0.0937 (7)	0.6450 (2)	0.5270 (2)	0.0861 (10)
H13A	-0.0207	0.6687	0.5773	0.103*
H13B	-0.0054	0.5970	0.5198	0.103*
H13C	-0.2768	0.6353	0.5289	0.103*
C14	-0.2972 (5)	0.96625 (16)	0.39423 (16)	0.0607 (7)
H14A	-0.2435	0.9131	0.3945	0.073*
H14B	-0.4683	0.9712	0.3644	0.073*
H14C	-0.3021	0.9836	0.4495	0.073*
C15	0.2180 (6)	1.02526 (17)	0.1559 (2)	0.0785 (9)
H15A	0.0714	1.0027	0.1228	0.094*
H15B	0.3626	0.9898	0.1603	0.094*
H15C	0.2675	1.0724	0.1309	0.094*
N1	-0.1189 (3)	0.92553 (10)	0.23762 (11)	0.0406 (4)
H1N	-0.281 (2)	0.9160 (14)	0.2363 (14)	0.049*
O1	0.3230 (3)	0.86960 (9)	0.24338 (10)	0.0492 (4)
O2	-0.0551 (3)	0.80389 (9)	0.16625 (10)	0.0554 (4)
S1	0.05387 (10)	0.84674 (3)	0.23607 (3)	0.03851 (18)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0356 (11)	0.0335 (10)	0.0442 (11)	0.0020 (9)	0.0100 (8)	-0.0017 (9)
C2	0.0488 (13)	0.0495 (13)	0.0513 (13)	-0.0121 (11)	0.0052 (10)	0.0000 (11)
C3	0.0612 (15)	0.0641 (15)	0.0454 (13)	-0.0033 (13)	0.0033 (11)	0.0023 (12)
C4	0.0602 (15)	0.0513 (14)	0.0529 (14)	0.0018 (12)	0.0212 (11)	0.0051 (11)
C5	0.0527 (14)	0.0510 (14)	0.0727 (17)	-0.0149 (12)	0.0175 (12)	0.0064 (13)
C6	0.0434 (12)	0.0475 (12)	0.0550 (14)	-0.0075 (11)	0.0049 (10)	0.0010 (11)
C7	0.0352 (11)	0.0332 (10)	0.0554 (13)	0.0062 (9)	0.0011 (9)	0.0055 (9)
C8	0.0462 (13)	0.0463 (13)	0.0551 (14)	0.0088 (11)	-0.0037 (10)	-0.0043 (11)
C9	0.081 (2)	0.0580 (16)	0.080 (2)	0.0120 (16)	-0.0092 (16)	-0.0198 (15)
C10	0.090 (2)	0.0443 (16)	0.132 (3)	-0.0081 (16)	-0.018 (2)	-0.0160 (19)
C11	0.0689 (19)	0.0416 (15)	0.136 (3)	-0.0097 (14)	0.0094 (19)	0.0171 (18)
C12	0.0478 (14)	0.0377 (12)	0.0872 (19)	0.0043 (11)	0.0088 (12)	0.0190 (12)
C13	0.103 (2)	0.090 (2)	0.0696 (19)	-0.0100 (19)	0.0280 (17)	0.0248 (17)
C14	0.0623 (16)	0.0715 (17)	0.0504 (14)	0.0100 (13)	0.0157 (11)	-0.0022 (13)
C15	0.082 (2)	0.0637 (17)	0.098 (2)	0.0144 (15)	0.0422 (17)	0.0360 (17)
N1	0.0308 (8)	0.0399 (9)	0.0509 (10)	-0.0004 (8)	0.0039 (8)	0.0021 (8)
O1	0.0343 (8)	0.0494 (9)	0.0662 (10)	0.0025 (7)	0.0162 (7)	0.0091 (8)
O2	0.0681 (11)	0.0544 (10)	0.0442 (9)	-0.0033 (8)	0.0087 (7)	-0.0100 (8)
S1	0.0364 (3)	0.0375 (3)	0.0431 (3)	0.0002 (2)	0.0108 (2)	-0.0002 (2)

Geometric parameters (Å, °)

C1—C6	1.374 (3)	C10—C11	1.362 (5)
C1—C2	1.381 (3)	C10—H10	0.9300
C1—S1	1.763 (2)	C11—C12	1.395 (4)
C2—C3	1.382 (3)	C11—H11	0.9300
C2—H2	0.9300	C12—C15	1.495 (4)
C3—C4	1.377 (3)	C13—H13A	0.9600
C3—H3	0.9300	C13—H13B	0.9600
C4—C5	1.384 (4)	C13—H13C	0.9600
C4—C13	1.503 (4)	C14—H14A	0.9600
C5—C6	1.374 (3)	C14—H14B	0.9600
C5—H5	0.9300	C14—H14C	0.9600
C6—H6	0.9300	C15—H15A	0.9600
C7—C8	1.391 (3)	C15—H15B	0.9600
C7—C12	1.397 (3)	C15—H15C	0.9600
C7—N1	1.440 (3)	N1—S1	1.6294 (18)
C8—C9	1.387 (4)	N1—H1N	0.848 (10)
C8—C14	1.507 (3)	O1—S1	1.4308 (15)
C9—C10	1.375 (5)	O2—S1	1.4251 (16)
C9—H9	0.9300		
C6—C1—C2	120.4 (2)	C12—C11—H11	119.2
C6—C1—S1	119.08 (17)	C11—C12—C7	117.1 (3)
C2—C1—S1	120.50 (16)	C11—C12—C15	119.8 (3)
C1—C2—C3	119.1 (2)	C7—C12—C15	123.1 (2)
C1—C2—H2	120.5	C4—C13—H13A	109.5
C3—C2—H2	120.5	C4—C13—H13B	109.5
C4—C3—C2	121.6 (2)	H13A—C13—H13B	109.5
C4—C3—H3	119.2	C4—C13—H13C	109.5
C2—C3—H3	119.2	H13A—C13—H13C	109.5
C3—C4—C5	117.9 (2)	H13B—C13—H13C	109.5
C3—C4—C13	121.8 (3)	C8—C14—H14A	109.5
C5—C4—C13	120.3 (2)	C8—C14—H14B	109.5
C6—C5—C4	121.6 (2)	H14A—C14—H14B	109.5
C6—C5—H5	119.2	C8—C14—H14C	109.5
C4—C5—H5	119.2	H14A—C14—H14C	109.5
C1—C6—C5	119.4 (2)	H14B—C14—H14C	109.5
C1—C6—H6	120.3	C12—C15—H15A	109.5
C5—C6—H6	120.3	C12—C15—H15B	109.5
C8—C7—C12	122.2 (2)	H15A—C15—H15B	109.5
C8—C7—N1	119.88 (19)	C12—C15—H15C	109.5
C12—C7—N1	117.8 (2)	H15A—C15—H15C	109.5
C9—C8—C7	117.8 (3)	H15B—C15—H15C	109.5
C9—C8—C14	119.8 (2)	C7—N1—S1	123.04 (13)
C7—C8—C14	122.4 (2)	C7—N1—H1N	117.4 (16)
C10—C9—C8	121.1 (3)	S1—N1—H1N	111.9 (17)
C10—C9—H9	119.5	O2—S1—O1	119.85 (10)

C8—C9—H9	119.5	O2—S1—N1	106.49 (10)
C11—C10—C9	120.1 (3)	O1—S1—N1	106.91 (9)
C11—C10—H10	119.9	O2—S1—C1	106.76 (10)
C9—C10—H10	119.9	O1—S1—C1	107.68 (10)
C10—C11—C12	121.6 (3)	N1—S1—C1	108.79 (9)
C10—C11—H11	119.2		
C6—C1—C2—C3	0.5 (3)	C10—C11—C12—C7	-2.6 (4)
S1—C1—C2—C3	-177.81 (18)	C10—C11—C12—C15	176.0 (3)
C1—C2—C3—C4	0.3 (4)	C8—C7—C12—C11	3.8 (3)
C2—C3—C4—C5	-1.3 (4)	N1—C7—C12—C11	-179.8 (2)
C2—C3—C4—C13	177.5 (3)	C8—C7—C12—C15	-174.7 (2)
C3—C4—C5—C6	1.5 (4)	N1—C7—C12—C15	1.7 (3)
C13—C4—C5—C6	-177.3 (3)	C8—C7—N1—S1	-103.9 (2)
C2—C1—C6—C5	-0.3 (3)	C12—C7—N1—S1	79.6 (2)
S1—C1—C6—C5	178.06 (18)	C7—N1—S1—O2	-157.30 (17)
C4—C5—C6—C1	-0.8 (4)	C7—N1—S1—O1	-28.06 (19)
C12—C7—C8—C9	-2.0 (3)	C7—N1—S1—C1	87.97 (18)
N1—C7—C8—C9	-178.4 (2)	C6—C1—S1—O2	-26.8 (2)
C12—C7—C8—C14	176.2 (2)	C2—C1—S1—O2	151.58 (18)
N1—C7—C8—C14	-0.2 (3)	C6—C1—S1—O1	-156.66 (17)
C7—C8—C9—C10	-1.1 (4)	C2—C1—S1—O1	21.7 (2)
C14—C8—C9—C10	-179.3 (3)	C6—C1—S1—N1	87.81 (19)
C8—C9—C10—C11	2.3 (5)	C2—C1—S1—N1	-93.86 (19)
C9—C10—C11—C12	-0.4 (5)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N \cdots O1 ⁱ	0.85 (1)	2.20 (1)	3.040 (2)	169 (2)

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