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2,4-Dimethyl-*N*-(2-methylphenyl)-benzenesulfonamide

P. G. Nirmala,[a] B. Thimme Gowda,[a]* Sabine Foro[b] and Hartmut Fuess[b]

[a]Department of Chemistry, Mangalore University, Mangalagangotri 574 199, Mangalore, India, and [b]Institute of Materials Science, Darmstadt University of Technology, Petersenstrasse 23, D-64287 Darmstadt, Germany
Correspondence e-mail: gowdabt@yahoo.com

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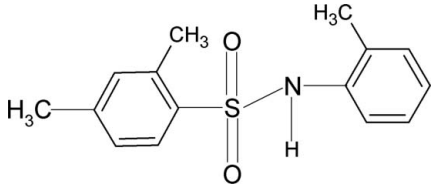
Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.003 Å; R factor = 0.039; wR factor = 0.116; data-to-parameter ratio = 16.1.

In the title compound, $C_{15}H_{17}NO_2S$, the molecule is bent at the S atom with a C—SO_2—NH—C torsion angle of 71.6 (1)°. The two benzene rings are tilted by 47.0 (1)° relative to each other. The crystal structure features inversion-related dimers linked by pairs of N—H···O hydrogen bonds.

Related literature

For the preparation of the title compound, see: Savitha & Gowda (2006). For related structures, see: Gelbrich *et al.* (2007); Gowda *et al.* (2008; 2009*a*,*b*); Perlovich *et al.* (2006).



Experimental

Crystal data

$C_{15}H_{17}NO_2S$
M_r = 275.36
Triclinic, $P\overline{1}$
a = 8.1789 (8) Å
b = 8.2659 (9) Å
c = 11.005 (1) Å
α = 96.249 (9)°
β = 96.078 (9)°
γ = 106.782 (9)°
V = 700.68 (12) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 299 K
0.48 × 0.26 × 0.12 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.898, T_{max} = 0.973
4894 measured reflections
2862 independent reflections
2446 reflections with $I > 2\sigma(I)$
R_{int} = 0.010

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.116
S = 1.05
2862 reflections
178 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O1^{i}	0.83 (2)	2.19 (2)	3.002 (2)	165 (2)

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

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: CI2971).

References

Gelbrich, T., Hursthouse, M. B. & Threlfall, T. L. (2007). *Acta Cryst.* B**63**, 621–632.
Gowda, B. T., Foro, S., Babitha, K. S. & Fuess, H. (2008). *Acta Cryst.* E**64**, o1692.
Gowda, B. T., Foro, S., Nirmala, P. G., Babitha, K. S. & Fuess, H. (2009*a*). *Acta Cryst.* E**65**, o576.
Gowda, B. T., Foro, S., Nirmala, P. G., Terao, H. & Fuess, H. (2009*b*). *Acta Cryst.* E**65**, o800.
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.* E**62**, o780–o782.
Savitha, M. B. & Gowda, B. T. (2006). *Z. Naturforsch. Teil A*, **60**, 600–606.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supporting information

Acta Cryst. (2009). E**65**, o3210 [doi:10.1107/S1600536809049563]

2,4-Dimethyl-*N*-(2-methylphenyl)benzenesulfonamide

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

S1. Comment

As part of a study of the substituent effects on the structures of *N*-(aryl)-arylsulfonamides (Gowda *et al.*, 2008; 2009*a,b*), in the present work, the structure of 2,4-dimethyl-*N*-(2-methylphenyl)benzenesulfonamide (I) has been determined (Fig. 1). The molecule is bent at the S atom with the C—SO_2—NH—C torsion angle of 71.6 (1)°, compared with the values of 73.0 (2)° in 4-chloro-2-methyl-*N*-(2-methylphenyl)benzenesulfonamide (II) (Gowda *et al.* 2009*b*), -46.1 (3)° (molecule 1) and 47.7 (3)° (molecule 2) in the two molecules of 2,4-dimethyl-*N*-(phenyl)benzenesulfonamide (III) (Gowda *et al.* 2009*a*) and 72.0 (2)° in *N*-(2-methylphenyl)benzenesulfonamide (IV) (Gowda *et al.*, 2008). The two benzene rings in (I) are tilted relative to each other by 47.0 (1)°, compared to the values of 45.8 (1)° in (II), 67.5 (1)° in molecule 1 and 72.9 (1)° in molecule 2 of (III), and 61.5 (1)° in (IV). The other bond parameters in (I) are similar to those observed in (II), (III), (IV) and other aryl sulfonamides(Perlovich *et al.*, 2006; Gelbrich *et al.*, 2007). The crystal packing of molecules in (I) is characterized by N—H···O hydrogen bonds (Table 1, Fig.2).

S2. Experimental

A solution of 1,3-xylene (1,3-dimethylbenzene) (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 2,4-dimethylbenzenesulfonylchloride was treated with *o*-toluidine 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 solid 2,4-dimethyl-*N*-(2-methylphenyl)benzenesulfonamide 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 (Savitha & Gowda, 2006). 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 its position was refined with the N-H distance restrained to 0.86 (2) Å. The other H atoms were positioned with idealized geometry using a riding model [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).

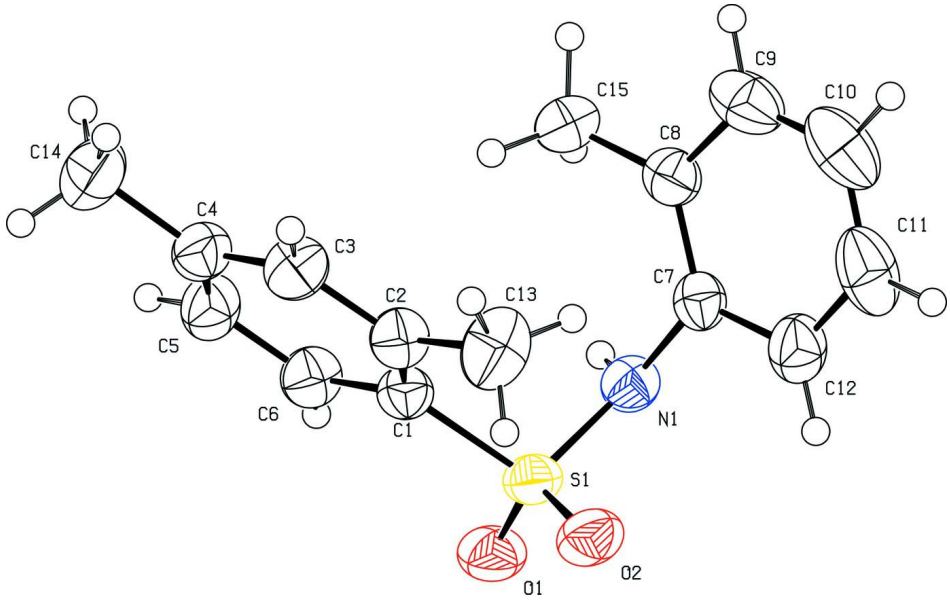


Figure 1

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

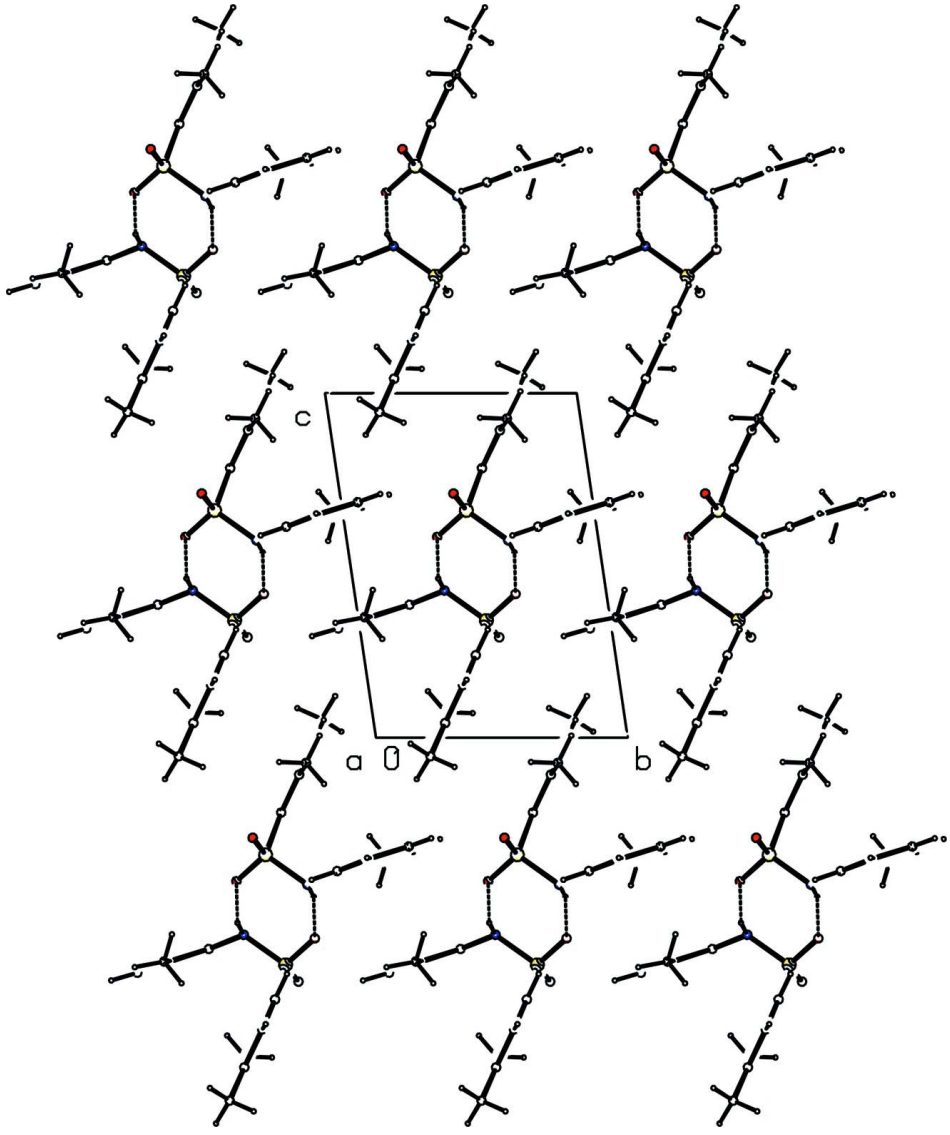


Figure 2

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

2,4-Dimethyl-*N*-(2-methylphenyl)benzenesulfonamide

Crystal data

$C_{15}H_{17}NO_2S$
$M_r = 275.36$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.1789\ (8)$ Å
$b = 8.2659\ (9)$ Å
$c = 11.005\ (1)$ Å
$\alpha = 96.249\ (9)°$
$\beta = 96.078\ (9)°$
$\gamma = 106.782\ (9)°$
$V = 700.68\ (12)$ Å^3
$Z = 2$
$F(000) = 292$
$D_x = 1.305$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2978 reflections
$\theta = 3.0–27.9°$
$\mu = 0.23$ mm^{-1}
$T = 299$ K
Prism, colourless
0.48 × 0.26 × 0.12 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} = 0.898, T_{max} = 0.973

4894 measured reflections
2862 independent reflections
2446 reflections with $I > 2\sigma(I)$
R_{int} = 0.010
θ_{max} = 26.4°, θ_{min} = 3.0°
h = −10→10
k = −10→7
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.116
S = 1.05
2862 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
$w = 1/[\sigma^2(F_o^2) + (0.064P)^2 + 0.2128P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
C1	0.1125 (2)	0.5794 (2)	0.78239 (16)	0.0390 (4)
C2	0.2116 (2)	0.6728 (2)	0.89249 (16)	0.0440 (4)
C3	0.1243 (3)	0.7397 (3)	0.97740 (17)	0.0496 (4)
H3	0.1876	0.8030	1.0513	0.060*
C4	−0.0514 (3)	0.7168 (2)	0.95762 (18)	0.0480 (4)
C5	−0.1451 (2)	0.6234 (3)	0.84761 (18)	0.0505 (4)
H5	−0.2633	0.6063	0.8322	0.061*
C6	−0.0645 (2)	0.5551 (2)	0.76046 (17)	0.0458 (4)
H6	−0.1287	0.4925	0.6866	0.055*
C7	0.4102 (2)	0.7886 (2)	0.61387 (14)	0.0380 (4)
C8	0.3900 (2)	0.9481 (2)	0.64940 (15)	0.0432 (4)
C9	0.5397 (3)	1.0860 (3)	0.68291 (19)	0.0599 (5)
H9	0.5304	1.1944	0.7053	0.072*
C10	0.7000 (3)	1.0659 (3)	0.6837 (2)	0.0693 (7)
H10	0.7978	1.1600	0.7075	0.083*

C11	0.7175 (3)	0.9075 (3)	0.6495 (2)	0.0691 (7)
H11	0.8268	0.8942	0.6511	0.083*
C12	0.5722 (2)	0.7676 (3)	0.61251 (19)	0.0525 (5)
H12	0.5831	0.6607	0.5870	0.063*
C13	0.4036 (3)	0.7076 (3)	0.9262 (2)	0.0642 (6)
H13A	0.4627	0.7664	0.8654	0.077*
H13B	0.4287	0.6014	0.9290	0.077*
H13C	0.4416	0.7771	1.0057	0.077*
C14	−0.1363 (3)	0.7946 (3)	1.0542 (2)	0.0660 (6)
H14A	−0.0497	0.8623	1.1206	0.079*
H14B	−0.2169	0.7053	1.0855	0.079*
H14C	−0.1960	0.8657	1.0176	0.079*
C15	0.2154 (3)	0.9733 (2)	0.6503 (2)	0.0583 (5)
H15A	0.1600	0.9167	0.7135	0.070*
H15B	0.1462	0.9261	0.5714	0.070*
H15C	0.2283	1.0932	0.6667	0.070*
N1	0.26247 (19)	0.64126 (19)	0.57272 (13)	0.0409 (3)
H1N	0.175 (2)	0.659 (3)	0.5378 (18)	0.049*
O1	0.06289 (17)	0.36111 (16)	0.58290 (13)	0.0527 (3)
O2	0.35102 (17)	0.45144 (17)	0.70879 (13)	0.0524 (3)
S1	0.20156 (5)	0.49271 (5)	0.65960 (4)	0.04048 (16)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0394 (9)	0.0368 (8)	0.0420 (9)	0.0129 (7)	0.0056 (7)	0.0065 (7)
C2	0.0413 (9)	0.0472 (9)	0.0434 (9)	0.0139 (8)	0.0035 (7)	0.0074 (7)
C3	0.0527 (11)	0.0531 (11)	0.0422 (9)	0.0177 (9)	0.0034 (8)	0.0025 (8)
C4	0.0538 (11)	0.0481 (10)	0.0500 (10)	0.0232 (9)	0.0146 (8)	0.0127 (8)
C5	0.0404 (9)	0.0584 (11)	0.0571 (11)	0.0200 (9)	0.0087 (8)	0.0123 (9)
C6	0.0412 (9)	0.0480 (10)	0.0473 (10)	0.0143 (8)	0.0020 (7)	0.0051 (8)
C7	0.0374 (8)	0.0422 (9)	0.0328 (8)	0.0095 (7)	0.0055 (6)	0.0050 (6)
C8	0.0522 (10)	0.0408 (9)	0.0344 (8)	0.0100 (8)	0.0084 (7)	0.0046 (7)
C9	0.0737 (14)	0.0435 (10)	0.0498 (11)	0.0012 (10)	0.0025 (10)	0.0043 (8)
C10	0.0565 (13)	0.0683 (14)	0.0624 (13)	−0.0109 (11)	−0.0078 (10)	0.0199 (11)
C11	0.0368 (10)	0.0928 (18)	0.0765 (15)	0.0099 (11)	0.0044 (10)	0.0380 (13)
C12	0.0426 (10)	0.0627 (12)	0.0579 (11)	0.0201 (9)	0.0117 (8)	0.0176 (9)
C13	0.0440 (11)	0.0873 (16)	0.0544 (12)	0.0194 (11)	−0.0049 (9)	−0.0059 (11)
C14	0.0683 (14)	0.0747 (15)	0.0644 (13)	0.0333 (12)	0.0219 (11)	0.0065 (11)
C15	0.0692 (13)	0.0461 (11)	0.0683 (13)	0.0261 (10)	0.0246 (11)	0.0074 (9)
N1	0.0391 (8)	0.0405 (8)	0.0409 (8)	0.0117 (6)	0.0020 (6)	0.0006 (6)
O1	0.0516 (7)	0.0371 (6)	0.0622 (8)	0.0085 (6)	0.0031 (6)	−0.0050 (6)
O2	0.0499 (7)	0.0472 (7)	0.0660 (8)	0.0246 (6)	0.0075 (6)	0.0078 (6)
S1	0.0399 (2)	0.0329 (2)	0.0477 (3)	0.01217 (17)	0.00452 (18)	0.00091 (17)

Geometric parameters (Å, º)

C1—C6	1.394 (2)	C10—C11	1.375 (4)
C1—C2	1.396 (2)	C10—H10	0.93
C1—S1	1.7799 (17)	C11—C12	1.384 (3)
C2—C3	1.394 (3)	C11—H11	0.93
C2—C13	1.510 (2)	C12—H12	0.93
C3—C4	1.385 (3)	C13—H13A	0.96
C3—H3	0.93	C13—H13B	0.96
C4—C5	1.381 (3)	C13—H13C	0.96
C4—C14	1.513 (3)	C14—H14A	0.96
C5—C6	1.380 (3)	C14—H14B	0.96
C5—H5	0.93	C14—H14C	0.96
C6—H6	0.93	C15—H15A	0.96
C7—C12	1.386 (2)	C15—H15B	0.96
C7—C8	1.395 (2)	C15—H15C	0.96
C7—N1	1.436 (2)	N1—S1	1.6350 (16)
C8—C9	1.393 (3)	N1—H1N	0.832 (15)
C8—C15	1.503 (3)	O1—S1	1.4377 (13)
C9—C10	1.368 (3)	O2—S1	1.4299 (13)
C9—H9	0.93		

C6—C1—C2	120.83 (16)	C12—C11—H11	120.1
C6—C1—S1	115.77 (13)	C11—C12—C7	119.3 (2)
C2—C1—S1	123.34 (13)	C11—C12—H12	120.3
C3—C2—C1	116.59 (16)	C7—C12—H12	120.3
C3—C2—C13	117.89 (17)	C2—C13—H13A	109.5
C1—C2—C13	125.51 (17)	C2—C13—H13B	109.5
C4—C3—C2	123.53 (18)	H13A—C13—H13B	109.5
C4—C3—H3	118.2	C2—C13—H13C	109.5
C2—C3—H3	118.2	H13A—C13—H13C	109.5
C5—C4—C3	118.18 (17)	H13B—C13—H13C	109.5
C5—C4—C14	121.52 (18)	C4—C14—H14A	109.5
C3—C4—C14	120.30 (19)	C4—C14—H14B	109.5
C6—C5—C4	120.45 (17)	H14A—C14—H14B	109.5
C6—C5—H5	119.8	C4—C14—H14C	109.5
C4—C5—H5	119.8	H14A—C14—H14C	109.5
C5—C6—C1	120.42 (17)	H14B—C14—H14C	109.5
C5—C6—H6	119.8	C8—C15—H15A	109.5
C1—C6—H6	119.8	C8—C15—H15B	109.5
C12—C7—C8	121.51 (17)	H15A—C15—H15B	109.5
C12—C7—N1	117.73 (16)	C8—C15—H15C	109.5
C8—C7—N1	120.71 (15)	H15A—C15—H15C	109.5
C9—C8—C7	117.21 (18)	H15B—C15—H15C	109.5
C9—C8—C15	120.60 (18)	C7—N1—S1	121.46 (11)
C7—C8—C15	122.19 (16)	C7—N1—H1N	116.5 (14)
C10—C9—C8	121.6 (2)	S1—N1—H1N	108.7 (15)
C10—C9—H9	119.2	O2—S1—O1	118.89 (8)

C8—C9—H9	119.2	O2—S1—N1	108.05 (8)
C9—C10—C11	120.4 (2)	O1—S1—N1	104.94 (8)
C9—C10—H10	119.8	O2—S1—C1	109.57 (8)
C11—C10—H10	119.8	O1—S1—C1	107.40 (8)
C10—C11—C12	119.9 (2)	N1—S1—C1	107.42 (8)
C10—C11—H11	120.1		

C6—C1—C2—C3	−0.2 (3)	C15—C8—C9—C10	179.47 (19)
S1—C1—C2—C3	176.86 (13)	C8—C9—C10—C11	0.9 (3)
C6—C1—C2—C13	−179.88 (19)	C9—C10—C11—C12	0.8 (4)
S1—C1—C2—C13	−2.8 (3)	C10—C11—C12—C7	−1.8 (3)
C1—C2—C3—C4	0.4 (3)	C8—C7—C12—C11	1.2 (3)
C13—C2—C3—C4	−179.95 (19)	N1—C7—C12—C11	178.85 (17)
C2—C3—C4—C5	−0.3 (3)	C12—C7—N1—S1	75.97 (18)
C2—C3—C4—C14	−179.61 (19)	C8—C7—N1—S1	−106.39 (17)
C3—C4—C5—C6	0.0 (3)	C7—N1—S1—O2	−46.56 (15)
C14—C4—C5—C6	179.35 (18)	C7—N1—S1—O1	−174.35 (12)
C4—C5—C6—C1	0.1 (3)	C7—N1—S1—C1	71.57 (14)
C2—C1—C6—C5	0.0 (3)	C6—C1—S1—O2	−152.26 (13)
S1—C1—C6—C5	−177.30 (14)	C2—C1—S1—O2	30.51 (17)
C12—C7—C8—C9	0.3 (3)	C6—C1—S1—O1	−21.81 (16)
N1—C7—C8—C9	−177.20 (15)	C2—C1—S1—O1	160.95 (14)
C12—C7—C8—C15	179.46 (17)	C6—C1—S1—N1	90.60 (14)
N1—C7—C8—C15	1.9 (3)	C2—C1—S1—N1	−86.63 (16)
C7—C8—C9—C10	−1.4 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.83 (2)	2.19 (2)	3.002 (2)	165 (2)

Symmetry code: (i) −*x*, −*y*+1, −*z*+1.