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4-Chloro-*N*-(2,6-dimethylphenyl)-2-methylbenzenesulfonamide

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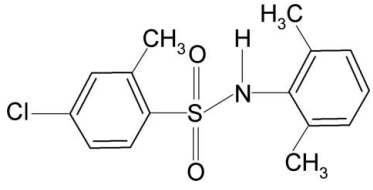
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.096; data-to-parameter ratio = 15.8.

In the title compound, $C_{15}H_{16}ClNO_2S$, the C—SO_2—NH—C torsion angle is −61.15 (16)°. The sulfonyl and aniline benzene rings are tilted relative to each other by 38.8 (1)°. 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 hydrogen-bonding modes of sulfonamides, see: Adsmond & Grant (2001). For studies on the effects of substituents on the structures and other aspects of *N*-(aryl)-amides, see: Gowda *et al.* (2000), on *N*-(aryl)-methanesulfonamides, see: Gowda *et al.* (2007), on *N*-(aryl)-arylsulfonamides, see: Gelbrich *et al.* (2007); Perlovich *et al.* (2006); Rodrigues *et al.* (2011); Shetty & Gowda (2005) and on *N*-(chloro)-arylsulfonamides, see: Gowda *et al.* (2003).



Experimental

Crystal data

$C_{15}H_{16}ClNO_2S$
M_r = 309.80
Triclinic, $P\overline{1}$
a = 8.275 (2) Å
b = 8.430 (2) Å
c = 11.195 (2) Å
α = 92.12 (1)°
β = 96.15 (1)°
γ = 109.58 (2)°
V = 729.3 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.41 mm^{-1}
T = 293 K
0.46 × 0.40 × 0.24 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.836, T_{max} = 0.909
4886 measured reflections
2948 independent reflections
2588 reflections with $I > 2\sigma(I)$
R_{int} = 0.011

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.096
S = 1.06
2948 reflections
187 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O2^{i}	0.84 (2)	2.21 (2)	3.024 (2)	165 (2)

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

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5663).

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supporting information

Acta Cryst. (2011). E**67**, o2890 [doi:10.1107/S1600536811040888]

4-Chloro-*N*-(2,6-dimethylphenyl)-2-methylbenzenesulfonamide

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S1. Comment

The amide and sulfonamide moieties are the constituents of many biologically significant compounds. The hydrogen bonding preferences of sulfonamides have been investigated (Adsmond & Grant, 2001). As part of our studies on the substituent effects on the structures and other aspects of *N*-(aryl)-amides (Gowda *et al.*, 2000), *N*-(aryl)-methanesulfonamides (Gowda *et al.*, 2007), *N*-(aryl)-arylsulfonamides (Rodrigues *et al.*, 2011; Shetty & Gowda, 2005) and *N*-(chloro)-arylsulfonamides (Gowda *et al.*, 2003), in the present work, the crystal structure of 4-Chloro-2-methyl-*N*-(2,6-dimethylphenyl)benzenesulfonamide (I) has been determined (Fig. 1).

In (I), the conformation of the N—C bond in the C—SO_2—NH—C segment has *gauche* torsions with respect to the S═O bonds. The molecule is bent at the S atom with the C—SO_2—NH—C torsion angle of -61.2 (2)°, compared to the value of 67.5 (2)° in 4-Chloro-2-methyl-*N*-(2,4-dimethylphenyl)benzenesulfonamide (II) (Rodrigues *et al.*, 2011).

The sulfonyl and the aniline benzene rings are tilted relative to each other by 38.8 (1)°, compared to the value of 44.5 (1)° in (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 intermolecular N–H···O hydrogen bonds (Table 1) link the molecules into dimeric chains. Part of the crystal structure is shown in Fig. 2.

S2. Experimental

The solution of *m*-chlorotoluene (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-methyl-4-chlorobenzenesulfonylchloride was treated with 2,6-dimethylaniline 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 cc). The resultant solid 4-chloro-2-methyl-*N*- (2,6-dimethylphenyl)-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).

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

S3. Refinement

The H atom of the NH group wwa located in a difference map and its coordinates were refined with the N-H distance restrained to 0.86 (2) %A. The other H atoms were positioned with idealized geometry using a riding model with the

aromatic C—H = 0.93Å and methyl C—H = 0.96 Å. All H atoms were refined with isotropic displacement parameters. The U_{iso}(H) values were set at 1.2U_{eq}(C-aromatic, N) and 1.5U_{eq}(C-methyl).

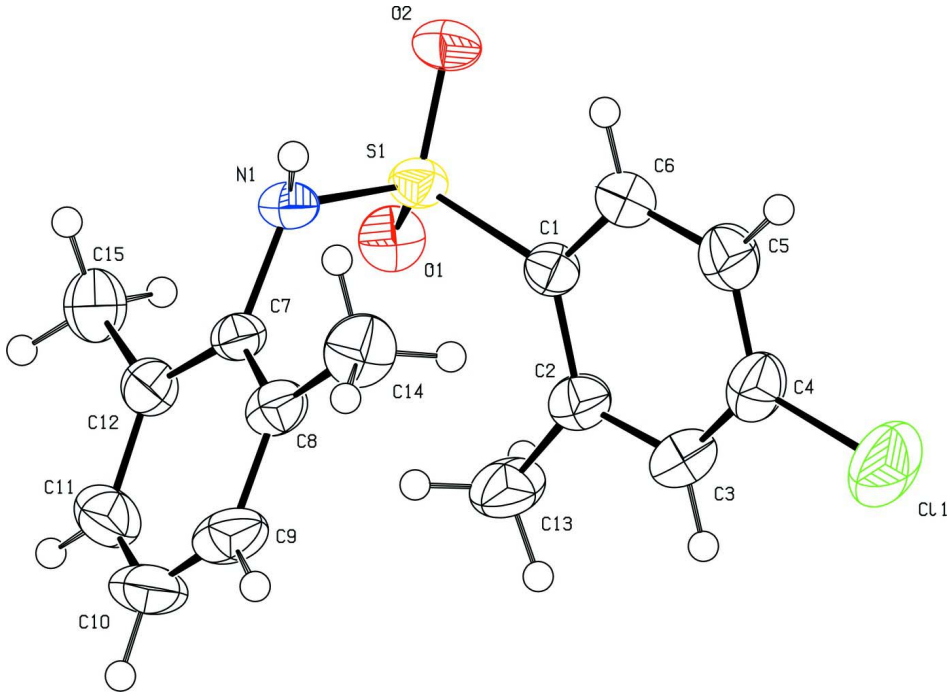

Figure 1

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

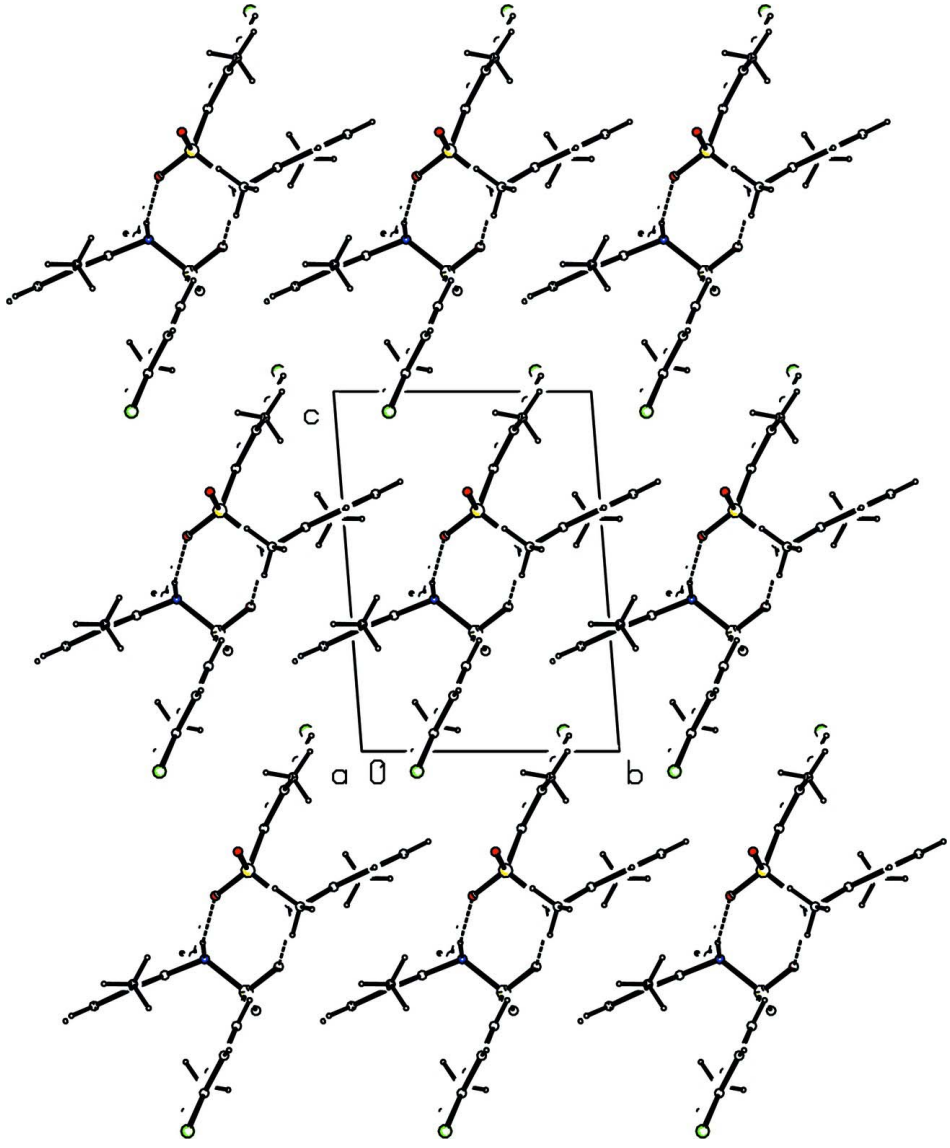

Figure 2

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

4-Chloro-*N*-(2,6-dimethylphenyl)-2-methylbenzenesulfonamide

Crystal data

$C_{15}H_{16}ClNO_2S$
$M_r = 309.80$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.275$ (2) Å
$b = 8.430$ (2) Å
$c = 11.195$ (2) Å
$\alpha = 92.12$ (1)°
$\beta = 96.15$ (1)°
$\gamma = 109.58$ (2)°
$V = 729.3$ (3) Å^3
$Z = 2$
$F(000) = 324$
$D_x = 1.411$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2916 reflections
$\theta = 2.6$–27.8°
$\mu = 0.41$ mm^{-1}
$T = 293$ K
Rod, colourless
0.46 × 0.40 × 0.24 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 scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.836, T_{max} = 0.909
4886 measured reflections
2948 independent reflections
2588 reflections with $I > 2\sigma(I)$
R_{int} = 0.011
θ_{max} = 26.4°, θ_{min} = 2.6°
$h = -9 \rightarrow 10$
$k = -10 \rightarrow 10$
$l = -13 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.096
S = 1.06
2948 reflections
187 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
$w = 1/[\sigma^2(F_o^2) + (0.0348P)^2 + 0.4969P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 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	0.3930 (2)	0.4028 (2)	0.21423 (16)	0.0332 (4)
C2	0.2952 (3)	0.3124 (2)	0.10740 (17)	0.0394 (4)
C3	0.3843 (3)	0.2532 (3)	0.02725 (18)	0.0467 (5)
H3	0.3235	0.1908	−0.0436	0.056*
C4	0.5591 (3)	0.2846 (3)	0.05010 (19)	0.0462 (5)
C5	0.6547 (3)	0.3732 (3)	0.1545 (2)	0.0462 (5)
H5	0.7730	0.3938	0.1694	0.055*
C6	0.5698 (2)	0.4307 (2)	0.23665 (18)	0.0395 (4)
H6	0.6318	0.4892	0.3084	0.047*
C7	0.1102 (2)	0.1667 (2)	0.37777 (15)	0.0314 (4)
C8	0.1613 (3)	0.0322 (2)	0.34000 (17)	0.0379 (4)
C9	0.0315 (3)	−0.1166 (3)	0.29221 (19)	0.0507 (6)
H9	0.0613	−0.2074	0.2649	0.061*
C10	−0.1402 (3)	−0.1317 (3)	0.2847 (2)	0.0569 (6)
H10	−0.2246	−0.2313	0.2505	0.068*

C11	−0.1880 (3)	−0.0012 (3)	0.3271 (2)	0.0514 (5)
H11	−0.3047	−0.0142	0.3226	0.062*
C12	−0.0641 (2)	0.1505 (2)	0.37689 (17)	0.0379 (4)
C13	0.1034 (3)	0.2753 (3)	0.0727 (2)	0.0559 (6)
H13A	0.0824	0.3789	0.0591	0.067*
H13B	0.0416	0.2222	0.1365	0.067*
H13C	0.0646	0.2012	0.0003	0.067*
C14	0.3469 (3)	0.0408 (3)	0.3529 (2)	0.0521 (5)
H14A	0.4067	0.1047	0.4268	0.063*
H14B	0.4007	0.0947	0.2862	0.063*
H14C	0.3516	−0.0714	0.3537	0.063*
C15	−0.1197 (3)	0.2859 (3)	0.4316 (2)	0.0492 (5)
H15A	−0.0981	0.3784	0.3810	0.059*
H15B	−0.0556	0.3249	0.5100	0.059*
H15C	−0.2410	0.2411	0.4387	0.059*
N1	0.2397 (2)	0.32745 (19)	0.42256 (14)	0.0335 (3)
H1N	0.325 (2)	0.324 (3)	0.4680 (18)	0.040*
O1	0.15174 (18)	0.51087 (17)	0.27909 (13)	0.0433 (3)
O2	0.43786 (18)	0.61202 (16)	0.40162 (13)	0.0448 (3)
Cl1	0.66049 (10)	0.20864 (10)	−0.05578 (6)	0.0746 (2)
S1	0.30108 (6)	0.47803 (5)	0.33118 (4)	0.03365 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0364 (9)	0.0281 (8)	0.0340 (9)	0.0096 (7)	0.0053 (7)	0.0015 (7)
C2	0.0418 (10)	0.0407 (10)	0.0341 (9)	0.0137 (8)	0.0006 (8)	0.0003 (8)
C3	0.0544 (13)	0.0512 (12)	0.0328 (10)	0.0182 (10)	0.0011 (9)	−0.0046 (9)
C4	0.0542 (12)	0.0479 (12)	0.0431 (11)	0.0229 (10)	0.0158 (9)	0.0044 (9)
C5	0.0382 (10)	0.0510 (12)	0.0497 (12)	0.0144 (9)	0.0093 (9)	0.0044 (9)
C6	0.0368 (10)	0.0360 (10)	0.0409 (10)	0.0071 (8)	0.0033 (8)	0.0000 (8)
C7	0.0365 (9)	0.0287 (9)	0.0253 (8)	0.0075 (7)	0.0003 (7)	−0.0003 (7)
C8	0.0501 (11)	0.0325 (9)	0.0313 (9)	0.0153 (8)	0.0030 (8)	0.0012 (7)
C9	0.0784 (16)	0.0300 (10)	0.0391 (11)	0.0152 (10)	0.0001 (10)	−0.0039 (8)
C10	0.0656 (15)	0.0368 (11)	0.0463 (12)	−0.0050 (10)	−0.0112 (11)	−0.0026 (9)
C11	0.0385 (11)	0.0539 (13)	0.0487 (12)	0.0010 (10)	−0.0030 (9)	0.0077 (10)
C12	0.0375 (10)	0.0398 (10)	0.0335 (9)	0.0096 (8)	0.0027 (8)	0.0055 (8)
C13	0.0439 (12)	0.0775 (16)	0.0412 (12)	0.0199 (11)	−0.0082 (9)	−0.0126 (11)
C14	0.0612 (14)	0.0455 (12)	0.0589 (14)	0.0290 (11)	0.0115 (11)	0.0042 (10)
C15	0.0445 (11)	0.0550 (13)	0.0540 (13)	0.0222 (10)	0.0133 (10)	0.0075 (10)
N1	0.0342 (8)	0.0305 (8)	0.0313 (8)	0.0078 (6)	−0.0030 (6)	−0.0029 (6)
O1	0.0454 (8)	0.0411 (8)	0.0487 (8)	0.0216 (6)	0.0057 (6)	0.0036 (6)
O2	0.0474 (8)	0.0298 (7)	0.0486 (8)	0.0042 (6)	0.0020 (6)	−0.0089 (6)
Cl1	0.0795 (5)	0.0927 (5)	0.0632 (4)	0.0401 (4)	0.0278 (3)	−0.0078 (4)
S1	0.0362 (2)	0.0266 (2)	0.0361 (2)	0.00905 (18)	0.00272 (18)	−0.00301 (17)

Geometric parameters (Å, º)

C1—C6	1.394 (3)	C10—C11	1.374 (4)
C1—C2	1.406 (3)	C10—H10	0.9300
C1—S1	1.7805 (19)	C11—C12	1.394 (3)
C2—C3	1.397 (3)	C11—H11	0.9300
C2—C13	1.515 (3)	C12—C15	1.500 (3)
C3—C4	1.374 (3)	C13—H13A	0.9600
C3—H3	0.9300	C13—H13B	0.9600
C4—C5	1.375 (3)	C13—H13C	0.9600
C4—Cl1	1.742 (2)	C14—H14A	0.9600
C5—C6	1.381 (3)	C14—H14B	0.9600
C5—H5	0.9300	C14—H14C	0.9600
C6—H6	0.9300	C15—H15A	0.9600
C7—C12	1.401 (3)	C15—H15B	0.9600
C7—C8	1.403 (3)	C15—H15C	0.9600
C7—N1	1.448 (2)	N1—S1	1.6405 (16)
C8—C9	1.392 (3)	N1—H1N	0.835 (15)
C8—C14	1.503 (3)	O1—S1	1.4250 (14)
C9—C10	1.377 (4)	O2—S1	1.4369 (14)
C9—H9	0.9300		

C6—C1—C2	120.62 (17)	C12—C11—H11	119.6
C6—C1—S1	115.99 (14)	C11—C12—C7	117.58 (19)
C2—C1—S1	123.31 (14)	C11—C12—C15	119.85 (19)
C3—C2—C1	116.59 (18)	C7—C12—C15	122.52 (18)
C3—C2—C13	117.95 (18)	C2—C13—H13A	109.5
C1—C2—C13	125.46 (18)	C2—C13—H13B	109.5
C4—C3—C2	121.87 (19)	H13A—C13—H13B	109.5
C4—C3—H3	119.1	C2—C13—H13C	109.5
C2—C3—H3	119.1	H13A—C13—H13C	109.5
C3—C4—C5	121.49 (19)	H13B—C13—H13C	109.5
C3—C4—Cl1	118.87 (17)	C8—C14—H14A	109.5
C5—C4—Cl1	119.64 (17)	C8—C14—H14B	109.5
C4—C5—C6	118.02 (19)	H14A—C14—H14B	109.5
C4—C5—H5	121.0	C8—C14—H14C	109.5
C6—C5—H5	121.0	H14A—C14—H14C	109.5
C5—C6—C1	121.38 (19)	H14B—C14—H14C	109.5
C5—C6—H6	119.3	C12—C15—H15A	109.5
C1—C6—H6	119.3	C12—C15—H15B	109.5
C12—C7—C8	122.19 (17)	H15A—C15—H15B	109.5
C12—C7—N1	117.89 (16)	C12—C15—H15C	109.5
C8—C7—N1	119.90 (16)	H15A—C15—H15C	109.5
C9—C8—C7	117.36 (19)	H15B—C15—H15C	109.5
C9—C8—C14	119.58 (19)	C7—N1—S1	119.87 (12)
C7—C8—C14	123.02 (17)	C7—N1—H1N	116.8 (15)
C10—C9—C8	121.1 (2)	S1—N1—H1N	109.8 (15)
C10—C9—H9	119.4	O1—S1—O2	118.97 (9)

C8—C9—H9	119.4	O1—S1—N1	108.26 (9)
C11—C10—C9	120.7 (2)	O2—S1—N1	105.13 (8)
C11—C10—H10	119.7	O1—S1—C1	108.66 (9)
C9—C10—H10	119.7	O2—S1—C1	107.46 (9)
C10—C11—C12	120.9 (2)	N1—S1—C1	107.89 (8)
C10—C11—H11	119.6		

C6—C1—C2—C3	0.2 (3)	C8—C9—C10—C11	−1.7 (3)
S1—C1—C2—C3	−176.35 (15)	C9—C10—C11—C12	1.1 (3)
C6—C1—C2—C13	−179.5 (2)	C10—C11—C12—C7	2.3 (3)
S1—C1—C2—C13	3.9 (3)	C10—C11—C12—C15	−175.1 (2)
C1—C2—C3—C4	−1.4 (3)	C8—C7—C12—C11	−5.4 (3)
C13—C2—C3—C4	178.4 (2)	N1—C7—C12—C11	176.31 (16)
C2—C3—C4—C5	1.3 (3)	C8—C7—C12—C15	171.99 (18)
C2—C3—C4—Cl1	−179.08 (17)	N1—C7—C12—C15	−6.3 (3)
C3—C4—C5—C6	−0.1 (3)	C12—C7—N1—S1	−86.30 (19)
Cl1—C4—C5—C6	−179.66 (16)	C8—C7—N1—S1	95.34 (18)
C4—C5—C6—C1	−1.1 (3)	C7—N1—S1—O1	56.27 (16)
C2—C1—C6—C5	1.0 (3)	C7—N1—S1—O2	−175.60 (13)
S1—C1—C6—C5	177.81 (16)	C7—N1—S1—C1	−61.15 (16)
C12—C7—C8—C9	4.8 (3)	C6—C1—S1—O1	154.04 (14)
N1—C7—C8—C9	−176.88 (16)	C2—C1—S1—O1	−29.23 (18)
C12—C7—C8—C14	−173.08 (18)	C6—C1—S1—O2	24.10 (17)
N1—C7—C8—C14	5.2 (3)	C2—C1—S1—O2	−159.17 (16)
C7—C8—C9—C10	−1.2 (3)	C6—C1—S1—N1	−88.80 (15)
C14—C8—C9—C10	176.7 (2)	C2—C1—S1—N1	87.93 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O2^{i}	0.84 (2)	2.21 (2)	3.024 (2)	165 (2)

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