Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Methyl-*N*-(2-methylbenzoyl)benzenesulfonamide

B. Thimme Gowda,[a]* Sabine Foro,[b] P. A. Suchetan[a] 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

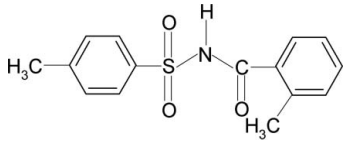
Received 21 May 2010; accepted 24 May 2010

Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.003 Å; R factor = 0.042; wR factor = 0.107; data-to-parameter ratio = 15.4.

In the title compound, $C_{15}H_{15}NO_3S$, the conformation of the N—H bond in the C—SO_2—NH—C(O) segment is *anti* to the C=O bond. Further, the conformation of the C=O bond is *syn* to the *ortho*-methyl group in the benzoyl ring. The dihedral angle between the sulfonyl benzene ring and the —SO_2—NH—C—O segment is 87.1 (1)° and that between the sulfonyl and the benzoyl benzene rings is 58.2 (1)°. In the crystal structure, molecules are linked by pairs of N—H···O(S) hydrogen bonds, forming inversion dimers.

Related literature

For background to our study of the effect of ring and side-chain substituents on the crystal structures of *N*-aromatic sulfonamides and for similar structures, see: Gowda *et al.* (2010*a*,*b*); Suchetan *et al.* (2010).



Experimental

Crystal data

$C_{15}H_{15}NO_3S$
M_r = 289.34
Triclinic, $P\overline{1}$
a = 6.4097 (8) Å
b = 10.433 (1) Å
c = 11.258 (1) Å
α = 79.17 (1)°
β = 74.34 (1)°
γ = 85.15 (2)°
V = 711.54 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 299 K
0.30 × 0.20 × 0.18 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.933, T_{max} = 0.959
4725 measured reflections
2872 independent reflections
2387 reflections with $I > 2\sigma(I)$
R_{int} = 0.013

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.107
S = 1.06
2872 reflections
186 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 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}	0.85 (1)	2.08 (1)	2.917 (2)	167 (2)

Symmetry code: (i) $-x+2, -y, -z$.

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*.

PAS thanks the Council of Scientific and Industrial Research (CSIR), Government of India, New Delhi, for the award of a research fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2213).

References

Gowda, B. T., Foro, S., Suchetan, P. A. & Fuess, H. (2010*a*). *Acta Cryst.* E**66**, o747.
Gowda, B. T., Foro, S., Suchetan, P. A. & Fuess, H. (2010*b*). *Acta Cryst.* E**66**, o1476.
Oxford Diffraction (2009). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Yarnton, ngland.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Suchetan, P. A., Gowda, B. T., Foro, S. & Fuess, H. (2010). *Acta Cryst.* E**66**, o1039.

supporting information

Acta Cryst. (2010). E**66**, o1502 [https://doi.org/10.1107/S1600536810019513]

4-Methyl-*N*-(2-methylbenzoyl)benzenesulfonamide

B. Thimme Gowda, Sabine Foro, P. A. Suchetan and Hartmut Fuess

S1. Comment

As a part of studying the effect of ring and the side chain substituents on the crystal structures of *N*-aromatic sulfonamides (Gowda *et al.*, 2010*a,b*; Suchetan *et al.*, 2010), the structure of *N*-(2-methylbenzoyl)-4-methylbenzene-sulfonamide (I) has been determined. The conformation of the N—H bond in the C—SO_2—NH—C(O) segment is *anti* to the C=O bond (Fig. 1), similar to those observed in *N*-(2-chlorobenzoyl)-4-chlorobenzenesulfonamide (II) (Gowda *et al.*, 2010*b*), *N*-(4-methylbenzoyl)-2-methylbenzenesulfonamide (III) (Gowda *et al.*, 2010*a*) and *N*-(benzoyl)-4-methyl-benzenesulfonamide (IV) (Suchetan *et al.*, 2010).

Further, the conformation of the C=O bond in the C—SO_2—NH—C(O) segment of (I) is *syn* to the *ortho*-methyl group in the benzoyl ring, similar to that observed between the C=O bond and the *ortho*-Cl in (II). The molecules are twisted at the *S* atom with the torsional angle of 67.7 (2)°, compared to those of 65.7 (2)° in (II), -53.1 (2)° and 61.2 (2)°, in the two independent molecules of (III) and 73.2 (2)° in (IV). The dihedral angle between the sulfonyl benzene ring and the —SO_2—NH—C—O segment is 87.1 (1)°, compared to the values of 88.5 (1)° in (II), 86.0 (1)° and 87.9 (1)° in the two molecules of (III) and 76.5 (1)° in (IV). Furthermore, the dihedral angle between the sulfonyl and the benzoyl benzene rings is 58.0 (1)°, compared to the values of 58.2 (1)° in (II), 88.1 (1)° (molecule 1) and 83.5 (1)° (molecule 2) in (III) and 79.4 (1)° in (IV). The packing of molecules linked by of N—H···O(S) hydrogen bonds (Table 1) is shown in Fig. 2.

S2. Experimental

The title compound was prepared by refluxing a mixture of 2-methylbenzoic acid, 4-methylbenzenesulfonamide and phosphorous oxy chloride for 3 h on a water bath. The resultant mixture was cooled and poured into ice cold water. The solid obtained was filtered, washed thoroughly with water and then dissolved in sodium bicarbonate solution. The compound was later reprecipitated by acidifying the filtered solution with dilute HCl. It was filtered, dried and recrystallized. Rod like colorless single crystals of the title compound used in X-ray diffraction studies were obtained by slow evaporation of its toluene solution 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) %A. 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).

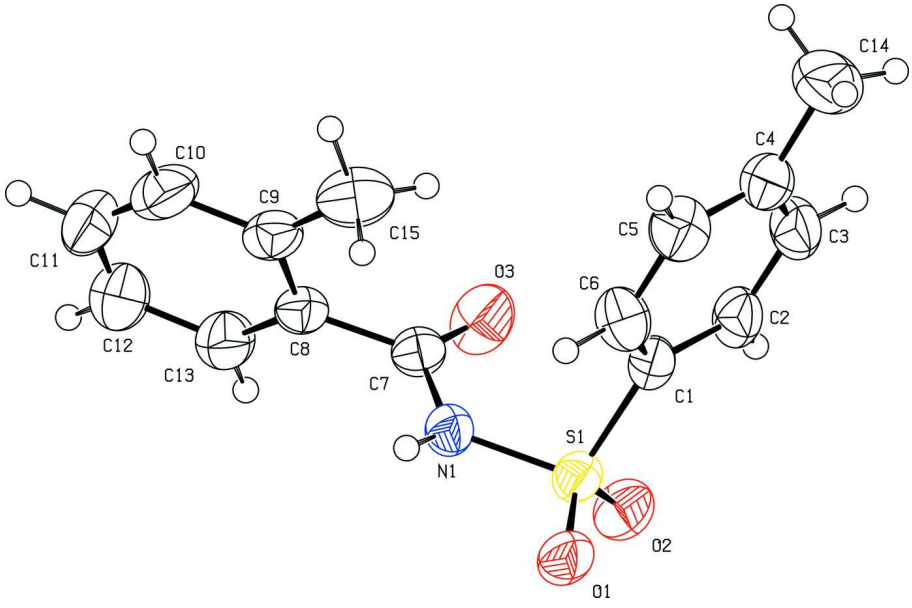


Figure 1

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

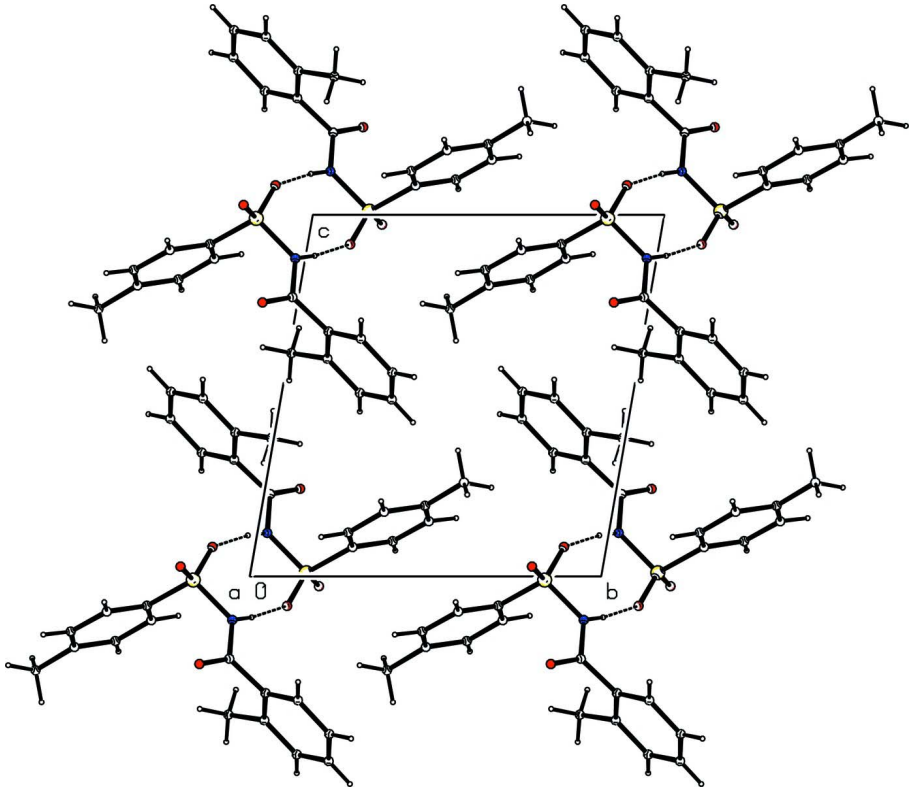


Figure 2

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

4-Methyl-*N*-(2-methylbenzoyl)benzenesulfonamide

Crystal data

$C_{15}H_{15}NO_3S$
$M_r = 289.34$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.4097$ (8) Å
$b = 10.433$ (1) Å
$c = 11.258$ (1) Å
$\alpha = 79.17$ (1)°
$\beta = 74.34$ (1)°
$\gamma = 85.15$ (2)°
$V = 711.54$ (13) Å^3
$Z = 2$
$F(000) = 304$
$D_x = 1.350$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2355 reflections
$\theta = 2.5$–27.8°
$\mu = 0.23$ mm^{-1}
$T = 299$ K
Rod, colorless
0.30 × 0.20 × 0.18 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)
$T_{min} = 0.933$, $T_{max} = 0.959$
4725 measured reflections
2872 independent reflections
2387 reflections with $I > 2\sigma(I)$
$R_{int} = 0.013$
$\theta_{max} = 26.4°$, $\theta_{min} = 2.5°$
$h = -8 \rightarrow 7$
$k = -9 \rightarrow 13$
$l = -13 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.107$
$S = 1.06$
2872 reflections
186 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
$w = 1/[\sigma^2(F_o^2) + (0.0434P)^2 + 0.3252P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.35$ e Å^{-3}

Special details

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

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}
S1	0.72322 (8)	0.15911 (5)	0.01004 (4)	0.04175 (15)
O1	0.9097 (2)	0.12090 (14)	−0.08160 (13)	0.0543 (4)
O2	0.5303 (2)	0.20184 (15)	−0.02716 (14)	0.0562 (4)
O3	0.3654 (2)	0.09970 (16)	0.24228 (16)	0.0646 (5)
N1	0.6788 (3)	0.02815 (15)	0.12042 (15)	0.0412 (4)
H1N	0.786 (2)	−0.0260 (17)	0.113 (2)	0.049*
C1	0.7964 (3)	0.27802 (18)	0.08116 (17)	0.0394 (4)
C2	0.6662 (3)	0.38915 (19)	0.0973 (2)	0.0487 (5)
H2	0.5389	0.4012	0.0714	0.058*
C3	0.7279 (4)	0.4821 (2)	0.1526 (2)	0.0589 (6)
H3	0.6434	0.5584	0.1612	0.071*
C4	0.9127 (4)	0.4638 (2)	0.1953 (2)	0.0548 (6)
C5	1.0396 (4)	0.3503 (2)	0.1788 (2)	0.0570 (6)
H5	1.1642	0.3365	0.2074	0.068*
C6	0.9848 (3)	0.2584 (2)	0.1211 (2)	0.0508 (5)
H6	1.0728	0.1840	0.1089	0.061*
C7	0.5092 (3)	0.01727 (19)	0.22766 (18)	0.0402 (4)
C8	0.5191 (3)	−0.10252 (19)	0.32282 (18)	0.0415 (4)
C9	0.6634 (3)	−0.1125 (2)	0.39775 (19)	0.0473 (5)
C10	0.6505 (4)	−0.2220 (3)	0.4924 (2)	0.0663 (7)
H10	0.7442	−0.2313	0.5440	0.080*
C11	0.5017 (5)	−0.3158 (3)	0.5106 (2)	0.0767 (8)
H11	0.4960	−0.3878	0.5741	0.092*
C12	0.3617 (5)	−0.3045 (3)	0.4360 (3)	0.0758 (8)
H12	0.2620	−0.3687	0.4486	0.091*
C13	0.3690 (4)	−0.1980 (2)	0.3425 (2)	0.0575 (6)
H13	0.2732	−0.1898	0.2923	0.069*
C14	0.9773 (5)	0.5629 (3)	0.2596 (3)	0.0849 (9)
H14A	0.8955	0.6432	0.2458	0.102*
H14B	1.1292	0.5782	0.2262	0.102*
H14C	0.9482	0.5302	0.3479	0.102*
C15	0.8194 (4)	−0.0075 (3)	0.3825 (2)	0.0658 (7)
H15A	0.9429	−0.0169	0.3136	0.079*
H15B	0.8659	−0.0150	0.4578	0.079*
H15C	0.7492	0.0765	0.3663	0.079*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0464 (3)	0.0408 (3)	0.0362 (3)	0.00360 (19)	−0.0112 (2)	−0.00359 (19)
O1	0.0642 (9)	0.0521 (8)	0.0364 (7)	0.0070 (7)	−0.0004 (7)	−0.0051 (6)
O2	0.0581 (9)	0.0609 (9)	0.0542 (9)	0.0045 (7)	−0.0287 (7)	−0.0036 (7)
O3	0.0505 (9)	0.0703 (11)	0.0600 (10)	0.0194 (8)	−0.0056 (8)	−0.0015 (8)
N1	0.0450 (9)	0.0362 (8)	0.0394 (9)	0.0031 (7)	−0.0082 (7)	−0.0052 (7)
C1	0.0388 (9)	0.0361 (9)	0.0386 (10)	0.0007 (7)	−0.0070 (8)	−0.0005 (8)

C2	0.0494 (11)	0.0409 (11)	0.0511 (12)	0.0089 (9)	−0.0124 (9)	−0.0017 (9)
C3	0.0708 (15)	0.0391 (11)	0.0573 (14)	0.0089 (10)	−0.0047 (12)	−0.0073 (10)
C4	0.0679 (14)	0.0458 (11)	0.0440 (12)	−0.0166 (10)	−0.0018 (10)	−0.0031 (9)
C5	0.0492 (12)	0.0588 (13)	0.0636 (14)	−0.0106 (10)	−0.0162 (11)	−0.0059 (11)
C6	0.0417 (11)	0.0451 (11)	0.0648 (14)	0.0037 (9)	−0.0139 (10)	−0.0101 (10)
C7	0.0387 (10)	0.0450 (10)	0.0393 (10)	−0.0024 (8)	−0.0130 (8)	−0.0083 (8)
C8	0.0446 (10)	0.0440 (10)	0.0345 (9)	0.0009 (8)	−0.0067 (8)	−0.0094 (8)
C9	0.0470 (11)	0.0563 (12)	0.0385 (10)	0.0101 (9)	−0.0110 (9)	−0.0129 (9)
C10	0.0794 (17)	0.0770 (17)	0.0417 (12)	0.0211 (14)	−0.0220 (12)	−0.0096 (11)
C11	0.111 (2)	0.0579 (15)	0.0474 (14)	0.0018 (15)	−0.0091 (15)	0.0068 (11)
C12	0.100 (2)	0.0588 (15)	0.0610 (16)	−0.0226 (14)	−0.0091 (15)	−0.0003 (12)
C13	0.0670 (14)	0.0553 (13)	0.0506 (13)	−0.0135 (11)	−0.0145 (11)	−0.0060 (10)
C14	0.115 (2)	0.0692 (17)	0.0694 (18)	−0.0326 (16)	−0.0078 (17)	−0.0204 (14)
C15	0.0528 (13)	0.0923 (19)	0.0612 (15)	−0.0041 (12)	−0.0248 (12)	−0.0202 (13)

Geometric parameters (Å, º)

S1—O2	1.4210 (15)	C7—C8	1.495 (3)
S1—O1	1.4351 (15)	C8—C13	1.390 (3)
S1—N1	1.6519 (17)	C8—C9	1.396 (3)
S1—C1	1.754 (2)	C9—C10	1.400 (3)
O3—C7	1.206 (2)	C9—C15	1.500 (3)
N1—C7	1.383 (2)	C10—C11	1.374 (4)
N1—H1N	0.849 (9)	C10—H10	0.9300
C1—C2	1.381 (3)	C11—C12	1.369 (4)
C1—C6	1.385 (3)	C11—H11	0.9300
C2—C3	1.381 (3)	C12—C13	1.374 (3)
C2—H2	0.9300	C12—H12	0.9300
C3—C4	1.381 (3)	C13—H13	0.9300
C3—H3	0.9300	C14—H14A	0.9600
C4—C5	1.392 (3)	C14—H14B	0.9600
C4—C14	1.509 (3)	C14—H14C	0.9600
C5—C6	1.371 (3)	C15—H15A	0.9600
C5—H5	0.9300	C15—H15B	0.9600
C6—H6	0.9300	C15—H15C	0.9600
O2—S1—O1	118.80 (9)	C13—C8—C9	120.8 (2)
O2—S1—N1	110.35 (9)	C13—C8—C7	118.47 (18)
O1—S1—N1	103.52 (8)	C9—C8—C7	120.44 (18)
O2—S1—C1	109.33 (9)	C8—C9—C10	117.4 (2)
O1—S1—C1	109.09 (9)	C8—C9—C15	121.8 (2)
N1—S1—C1	104.75 (9)	C10—C9—C15	120.7 (2)
C7—N1—S1	124.66 (14)	C11—C10—C9	121.2 (2)
C7—N1—H1N	121.5 (15)	C11—C10—H10	119.4
S1—N1—H1N	112.5 (15)	C9—C10—H10	119.4
C2—C1—C6	120.86 (19)	C12—C11—C10	120.6 (2)
C2—C1—S1	119.99 (16)	C12—C11—H11	119.7
C6—C1—S1	119.15 (15)	C10—C11—H11	119.7

C3—C2—C1	119.0 (2)	C11—C12—C13	119.8 (3)
C3—C2—H2	120.5	C11—C12—H12	120.1
C1—C2—H2	120.5	C13—C12—H12	120.1
C4—C3—C2	121.3 (2)	C12—C13—C8	120.2 (2)
C4—C3—H3	119.4	C12—C13—H13	119.9
C2—C3—H3	119.4	C8—C13—H13	119.9
C3—C4—C5	118.4 (2)	C4—C14—H14A	109.5
C3—C4—C14	121.6 (2)	C4—C14—H14B	109.5
C5—C4—C14	120.0 (2)	H14A—C14—H14B	109.5
C6—C5—C4	121.4 (2)	C4—C14—H14C	109.5
C6—C5—H5	119.3	H14A—C14—H14C	109.5
C4—C5—H5	119.3	H14B—C14—H14C	109.5
C5—C6—C1	119.06 (19)	C9—C15—H15A	109.5
C5—C6—H6	120.5	C9—C15—H15B	109.5
C1—C6—H6	120.5	H15A—C15—H15B	109.5
O3—C7—N1	121.68 (18)	C9—C15—H15C	109.5
O3—C7—C8	123.12 (18)	H15A—C15—H15C	109.5
N1—C7—C8	115.21 (16)	H15B—C15—H15C	109.5

O2—S1—N1—C7	−49.84 (18)	S1—C1—C6—C5	−178.79 (17)
O1—S1—N1—C7	−177.98 (16)	S1—N1—C7—O3	8.0 (3)
C1—S1—N1—C7	67.73 (17)	S1—N1—C7—C8	−171.57 (13)
O2—S1—C1—C2	2.47 (19)	O3—C7—C8—C13	70.2 (3)
O1—S1—C1—C2	133.91 (16)	N1—C7—C8—C13	−110.2 (2)
N1—S1—C1—C2	−115.79 (17)	O3—C7—C8—C9	−104.1 (2)
O2—S1—C1—C6	−177.85 (16)	N1—C7—C8—C9	75.4 (2)
O1—S1—C1—C6	−46.42 (18)	C13—C8—C9—C10	0.1 (3)
N1—S1—C1—C6	63.89 (18)	C7—C8—C9—C10	174.35 (18)
C6—C1—C2—C3	0.9 (3)	C13—C8—C9—C15	−176.9 (2)
S1—C1—C2—C3	−179.44 (16)	C7—C8—C9—C15	−2.7 (3)
C1—C2—C3—C4	−2.1 (3)	C8—C9—C10—C11	0.1 (3)
C2—C3—C4—C5	1.5 (3)	C15—C9—C10—C11	177.2 (2)
C2—C3—C4—C14	−177.9 (2)	C9—C10—C11—C12	0.0 (4)
C3—C4—C5—C6	0.3 (3)	C10—C11—C12—C13	−0.3 (4)
C14—C4—C5—C6	179.8 (2)	C11—C12—C13—C8	0.6 (4)
C4—C5—C6—C1	−1.5 (3)	C9—C8—C13—C12	−0.5 (3)
C2—C1—C6—C5	0.9 (3)	C7—C8—C13—C12	−174.8 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.85 (1)	2.08 (1)	2.917 (2)	167 (2)

Symmetry code: (i) −*x*+2, −*y*, −*z*.