Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

N-(3,5-Dimethylphenyl)benzenesulfonamide

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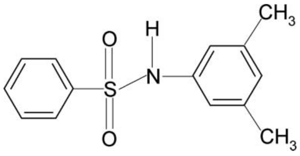
Received 15 November 2009; accepted 21 November 2009

Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.111; data-to-parameter ratio = 16.2.

In the crystal structure of the title compound, $C_{14}H_{15}NO_2S$, the molecule is bent at the S atom with a C—SO_2—NH—C torsion angle of 67.9 (2)°. The two benzene rings are tilted by 54.6 (1)° relative to each other. In the crystal, intermolecular N—H$\cdots$O hydrogen bonds pack the molecules into a supramolecular structure.

Related literature

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



Experimental

Crystal data

$C_{14}H_{15}NO_2S$
M_r = 261.33
Monoclinic, $P2_1/c$
a = 11.192 (1) Å
b = 7.3543 (7) Å
c = 16.672 (2) Å
β = 101.62 (1)°
V = 1344.1 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 299 K
0.48 × 0.40 × 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} = 0.896, T_{max} = 0.959
5063 measured reflections
2742 independent reflections
2187 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.111
S = 1.05
2742 reflections
169 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H$\cdots$$A$	D—H	H$\cdots$$A$	$D$$\cdots$$A$	D—H$\cdots$$A$
N1—H1$N$$\cdots$O1^{i}	0.81 (2)	2.14 (2)	2.942 (2)	176 (2)

Symmetry code: (i) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$.

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: BX2251).

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supporting information

Acta Cryst. (2009). E**65**, o3225 [doi:10.1107/S1600536809050089]

N-(3,5-Dimethylphenyl)benzenesulfonamide

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

S1. Comment

As part of a study of substituent effects on the structures of *N*-(aryl)-arylsulfonamides (Gowda *et al.*, 2008; 2009*a,b*), in the present work, the structure of *N*-(3,5-dimethylphenyl)benzenesulfonamide (I) has been determined (Fig. 1). The molecule is bent at the *S* atom with the C1—SO$_2$—NH—C7 torsion angle of 67.9 (2)°, compared to the values of 71.0 (2)° in *N*-(2,3-dimethylphenyl)benzenesulfonamide (II)(Gowda *et al.*, 2009*a*), 62.7 (2)° in *N*-(2,5-dimethylphenyl)-benzenesulfonamide (III) (Gowda *et al.*, 2009*b*) and -78.7 (2)° in *N*-(2,6-dimethylphenyl)benzenesulfonamide (IV) (Gowda *et al.*, 2008). The two benzene rings in (I) are tilted relative to each other by 54.6 (1)°, compared to the values of 64.8 (1)° in (II), 40.4 (1)° in (III) and 44.9 (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) *via* N—H···O(S) hydrogen bonds (Table 1) is shown in Fig.2.

S2. Experimental

The solution of benzene (10 cc) in chloroform (40 cc) was treated dropwise with chlorosulfonic acid (25 cc) 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 benzenesulfonylchloride was treated with 3,5-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 *N*-(3,5-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 (Gowda *et al.*, 2005). The 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 was located in difference map and its positional parameters were refined freely [N—H = 0.81 (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 $U_{\rm eq}$ of the parent atom).

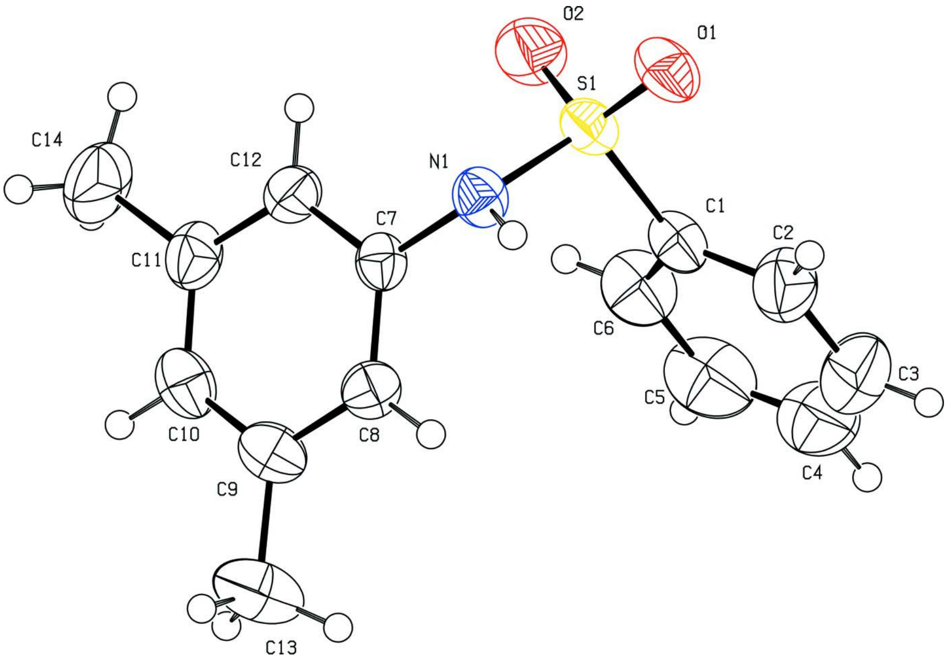

Figure 1

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

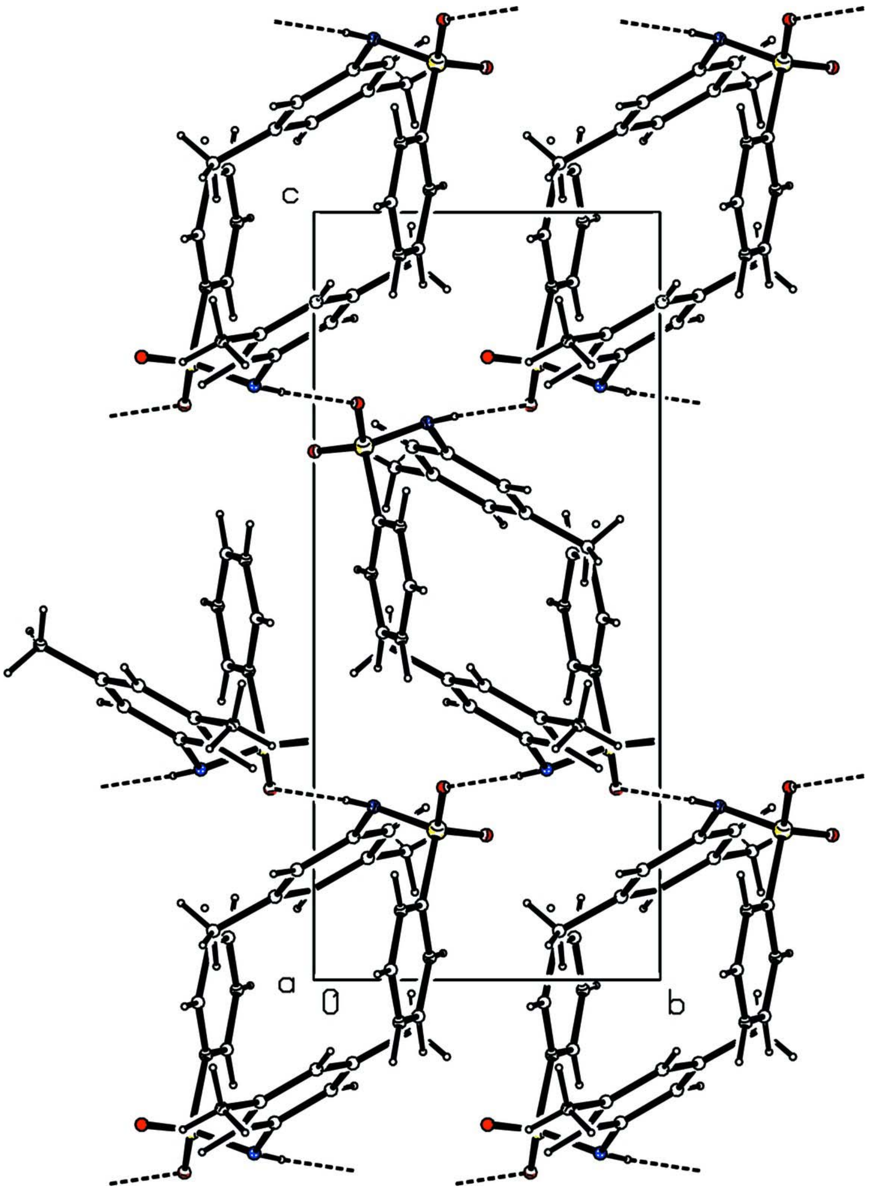

Figure 2

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

N-(3,5-Dimethylphenyl)benzenesulfonamide

Crystal data

$C_{14}H_{15}NO_2S$
$M_r = 261.33$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 11.192\ (1)$ Å
$b = 7.3543\ (7)$ Å
$c = 16.672\ (2)$ Å
$\beta = 101.62\ (1)°$
$V = 1344.1\ (2)$ Å^3
$Z = 4$

$F(000) = 552$
$D_x = 1.291$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 2.5$–$27.8°$
$\mu = 0.23$ mm^{-1}
$T = 299$ K
Prism, colourless
0.48 × 0.40 × 0.18 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector	5063 measured reflections
Radiation source: fine-focus sealed tube	2742 independent reflections
Graphite monochromator	2187 reflections with $I > 2\sigma(I)$
Rotation method data acquisition using ω and phi scans	$R_{\text{int}} = 0.015$
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)	$\theta_{\text{max}} = 26.4°$, $\theta_{\text{min}} = 2.5°$
$T_{\text{min}} = 0.896$, $T_{\text{max}} = 0.959$	$h = -13 \rightarrow 13$
	$k = -6 \rightarrow 9$
	$l = -9 \rightarrow 20$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.038$	$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 0.3783P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.111$	$(\Delta/\sigma)_{\text{max}} = 0.004$
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.31$ e Å^{-3}
2742 reflections	$\Delta\rho_{\text{min}} = -0.31$ e Å^{-3}
169 parameters	Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
0 restraints	Extinction coefficient: 0.0115 (18)
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.41245 (16)	0.1889 (3)	0.59746 (11)	0.0434 (4)
C2	0.52681 (19)	0.2478 (3)	0.59053 (14)	0.0618 (6)
H2	0.5873	0.2668	0.6369	0.074*
C3	0.5495 (3)	0.2780 (4)	0.51321 (17)	0.0816 (8)
H3	0.6266	0.3161	0.5074	0.098*
C4	0.4597 (3)	0.2524 (4)	0.44490 (15)	0.0790 (8)
H4	0.4764	0.2716	0.3931	0.095*
C5	0.3458 (3)	0.1986 (4)	0.45274 (14)	0.0805 (8)
H5	0.2846	0.1846	0.4063	0.097*
C6	0.3209 (2)	0.1650 (3)	0.52926 (13)	0.0620 (6)
H6	0.2438	0.1268	0.5347	0.074*
C7	0.20322 (14)	0.3865 (2)	0.68610 (10)	0.0354 (4)

C8	0.18940 (16)	0.5491 (2)	0.64340 (10)	0.0410 (4)
H8	0.2578	0.6162	0.6381	0.049*
C9	0.07428 (18)	0.6121 (3)	0.60856 (12)	0.0479 (5)
C10	−0.02579 (17)	0.5069 (3)	0.61670 (12)	0.0514 (5)
H10	−0.1035	0.5477	0.5929	0.062*
C11	−0.01416 (16)	0.3444 (3)	0.65873 (12)	0.0489 (5)
C12	0.10215 (16)	0.2850 (3)	0.69465 (11)	0.0430 (4)
H12	0.1122	0.1772	0.7244	0.052*
C13	0.0577 (2)	0.7909 (3)	0.56388 (16)	0.0733 (7)
H13A	0.1323	0.8239	0.5476	0.088*
H13B	0.0364	0.8830	0.5993	0.088*
H13C	−0.0063	0.7799	0.5162	0.088*
C14	−0.12452 (19)	0.2338 (4)	0.66755 (16)	0.0755 (7)
H14A	−0.1717	0.2058	0.6142	0.091*
H14B	−0.1735	0.3023	0.6978	0.091*
H14C	−0.0985	0.1229	0.6962	0.091*
N1	0.32271 (13)	0.3272 (2)	0.72571 (9)	0.0395 (4)
H1N	0.3739 (18)	0.406 (3)	0.7338 (12)	0.047*
O1	0.49861 (12)	0.12569 (19)	0.75071 (8)	0.0526 (4)
O2	0.29563 (13)	0.00239 (19)	0.68847 (10)	0.0590 (4)
S1	0.38310 (4)	0.14444 (6)	0.69537 (3)	0.04071 (17)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0437 (10)	0.0417 (10)	0.0431 (9)	0.0098 (8)	0.0043 (8)	−0.0040 (8)
C2	0.0500 (12)	0.0781 (15)	0.0577 (13)	0.0046 (11)	0.0119 (10)	0.0035 (11)
C3	0.0782 (18)	0.100 (2)	0.0758 (17)	0.0074 (16)	0.0366 (14)	0.0110 (16)
C4	0.110 (2)	0.0806 (18)	0.0512 (14)	0.0199 (16)	0.0283 (15)	0.0035 (12)
C5	0.104 (2)	0.0826 (18)	0.0466 (13)	0.0137 (16)	−0.0035 (13)	−0.0090 (12)
C6	0.0616 (14)	0.0681 (14)	0.0509 (12)	0.0016 (11)	−0.0019 (10)	−0.0081 (10)
C7	0.0323 (8)	0.0407 (9)	0.0330 (8)	0.0014 (7)	0.0063 (6)	−0.0033 (7)
C8	0.0411 (9)	0.0395 (9)	0.0419 (9)	−0.0038 (8)	0.0074 (7)	−0.0010 (7)
C9	0.0507 (11)	0.0427 (10)	0.0458 (10)	0.0040 (8)	−0.0008 (8)	−0.0007 (8)
C10	0.0365 (10)	0.0596 (12)	0.0539 (11)	0.0079 (9)	−0.0012 (8)	−0.0014 (10)
C11	0.0354 (9)	0.0621 (12)	0.0495 (11)	−0.0036 (8)	0.0093 (8)	0.0006 (9)
C12	0.0393 (9)	0.0463 (10)	0.0441 (10)	−0.0018 (8)	0.0101 (7)	0.0058 (8)
C13	0.0773 (16)	0.0504 (13)	0.0804 (17)	0.0035 (12)	−0.0119 (13)	0.0119 (12)
C14	0.0411 (12)	0.100 (2)	0.0858 (17)	−0.0124 (12)	0.0142 (11)	0.0160 (15)
N1	0.0326 (8)	0.0392 (8)	0.0450 (8)	−0.0012 (6)	0.0038 (6)	−0.0030 (6)
O1	0.0436 (7)	0.0577 (8)	0.0510 (7)	0.0142 (6)	−0.0032 (6)	0.0062 (6)
O2	0.0556 (8)	0.0410 (7)	0.0798 (10)	−0.0062 (6)	0.0123 (7)	−0.0002 (7)
S1	0.0367 (3)	0.0373 (3)	0.0457 (3)	0.00433 (18)	0.00246 (18)	0.00225 (18)

Geometric parameters (Å, º)

C1—C2	1.378 (3)	C9—C10	1.390 (3)
C1—C6	1.380 (3)	C9—C13	1.505 (3)

C1—S1	1.7594 (19)	C10—C11	1.378 (3)
C2—C3	1.381 (3)	C10—H10	0.9300
C2—H2	0.9300	C11—C12	1.389 (2)
C3—C4	1.372 (4)	C11—C14	1.511 (3)
C3—H3	0.9300	C12—H12	0.9300
C4—C5	1.366 (4)	C13—H13A	0.9600
C4—H4	0.9300	C13—H13B	0.9600
C5—C6	1.382 (3)	C13—H13C	0.9600
C5—H5	0.9300	C14—H14A	0.9600
C6—H6	0.9300	C14—H14B	0.9600
C7—C8	1.384 (2)	C14—H14C	0.9600
C7—C12	1.386 (2)	N1—S1	1.6302 (15)
C7—N1	1.435 (2)	N1—H1N	0.81 (2)
C8—C9	1.382 (2)	O1—S1	1.4356 (13)
C8—H8	0.9300	O2—S1	1.4204 (14)

C2—C1—C6	121.3 (2)	C9—C10—H10	118.8
C2—C1—S1	119.10 (15)	C10—C11—C12	118.44 (17)
C6—C1—S1	119.64 (16)	C10—C11—C14	121.40 (19)
C1—C2—C3	118.5 (2)	C12—C11—C14	120.15 (19)
C1—C2—H2	120.7	C7—C12—C11	119.97 (17)
C3—C2—H2	120.7	C7—C12—H12	120.0
C4—C3—C2	120.7 (2)	C11—C12—H12	120.0
C4—C3—H3	119.6	C9—C13—H13A	109.5
C2—C3—H3	119.6	C9—C13—H13B	109.5
C5—C4—C3	120.2 (2)	H13A—C13—H13B	109.5
C5—C4—H4	119.9	C9—C13—H13C	109.5
C3—C4—H4	119.9	H13A—C13—H13C	109.5
C4—C5—C6	120.4 (2)	H13B—C13—H13C	109.5
C4—C5—H5	119.8	C11—C14—H14A	109.5
C6—C5—H5	119.8	C11—C14—H14B	109.5
C1—C6—C5	118.9 (2)	H14A—C14—H14B	109.5
C1—C6—H6	120.5	C11—C14—H14C	109.5
C5—C6—H6	120.5	H14A—C14—H14C	109.5
C8—C7—C12	120.60 (16)	H14B—C14—H14C	109.5
C8—C7—N1	119.75 (15)	C7—N1—S1	120.92 (12)
C12—C7—N1	119.56 (16)	C7—N1—H1N	115.0 (15)
C9—C8—C7	120.24 (16)	S1—N1—H1N	108.7 (15)
C9—C8—H8	119.9	O2—S1—O1	119.88 (9)
C7—C8—H8	119.9	O2—S1—N1	108.03 (8)
C8—C9—C10	118.28 (18)	O1—S1—N1	104.82 (8)
C8—C9—C13	120.85 (19)	O2—S1—C1	108.42 (9)
C10—C9—C13	120.87 (18)	O1—S1—C1	107.56 (9)
C11—C10—C9	122.46 (17)	N1—S1—C1	107.53 (8)
C11—C10—H10	118.8		

C6—C1—C2—C3	−1.8 (3)	C8—C7—C12—C11	1.4 (3)
S1—C1—C2—C3	178.45 (19)	N1—C7—C12—C11	177.90 (16)

C1—C2—C3—C4	0.9 (4)	C10—C11—C12—C7	−1.5 (3)
C2—C3—C4—C5	0.8 (4)	C14—C11—C12—C7	179.70 (19)
C3—C4—C5—C6	−1.7 (4)	C8—C7—N1—S1	−114.44 (16)
C2—C1—C6—C5	0.9 (3)	C12—C7—N1—S1	69.0 (2)
S1—C1—C6—C5	−179.33 (19)	C7—N1—S1—O2	−48.89 (16)
C4—C5—C6—C1	0.9 (4)	C7—N1—S1—O1	−177.81 (13)
C12—C7—C8—C9	−0.1 (3)	C7—N1—S1—C1	67.93 (15)
N1—C7—C8—C9	−176.67 (16)	C2—C1—S1—O2	−148.57 (17)
C7—C8—C9—C10	−0.9 (3)	C6—C1—S1—O2	31.65 (19)
C7—C8—C9—C13	178.38 (19)	C2—C1—S1—O1	−17.56 (19)
C8—C9—C10—C11	0.7 (3)	C6—C1—S1—O1	162.66 (16)
C13—C9—C10—C11	−178.5 (2)	C2—C1—S1—N1	94.86 (17)
C9—C10—C11—C12	0.5 (3)	C6—C1—S1—N1	−84.93 (18)
C9—C10—C11—C14	179.3 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.81 (2)	2.14 (2)	2.942 (2)	176 (2)

Symmetry code: (i) −*x*+1, *y*+1/2, −*z*+3/2.