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N-(2-Methylphenyl)-2-nitrobenzenesulfonamide

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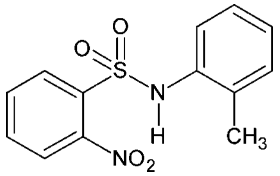
Key indicators: single-crystal X-ray study; *T* = 293 K; mean σ(C–C) = 0.005 Å; *R* factor = 0.053; *wR* factor = 0.114; data-to-parameter ratio = 13.6.

In the title compound, $C_{13}H_{12}N_2O_4S$, the dihedral angle between the benzene rings is 53.44 (14)°. The amide H atom exhibits bifurcated hydrogen bonding: an intramolecular N—H···O hydrogen bond generates an *S*(7) motif while in the crystal, N—H···O(S) hydrogen bonds link the molecules into zigzag *C*(4) chains along the *c* axis.

Related literature

For studies on the effects of substituents on the structures and other aspects of *N*-(aryl)-amides, see: Alkan *et al.* (2011); Bowes *et al.* (2003); Gowda & Weiss (1994); Saeed *et al.* (2010); Shahwar *et al.* (2012), of *N*-arylsulfonamides, see: Chaithanya *et al.* (2012); Gowda *et al.* (2002) and of *N*-chloroarylsulfonamides, see: Gowda & Shetty (2004); Shetty & Gowda (2004). For hydrogen-bonding patterns and motifs, see: Adsmond *et al.* (2001); Allen *et al.* (1998); Bernstein *et al.* (1995); Etter (1990).



Experimental

Crystal data

$C_{13}H_{12}N_2O_4S$
M_r = 292.31
Monoclinic, $P2_1/c$
a = 9.2409 (7) Å
b = 15.1531 (9) Å
c = 10.5376 (8) Å
β = 107.775 (8)°
V = 1405.13 (17) Å^3
Z = 4
Mo *K*α radiation
μ = 0.24 mm^{-1}
T = 293 K
0.30 × 0.28 × 0.14 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
$T_{\min}$ = 0.930, $T_{\max}$ = 0.967
5234 measured reflections
2538 independent reflections
2020 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.114
S = 1.21
2538 reflections
186 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ = 0.33 e Å^{-3}
$\Delta\rho_{\min}$ = −0.36 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$^{\rm i}$	0.84 (2)	2.16 (2)	2.897 (3)	146 (3)
N1—H1*N*···O3	0.84 (2)	2.57 (3)	3.132 (4)	125 (3)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2009); 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: BT5990).

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supporting information

Acta Cryst. (2012). E**68**, o2649   [doi:10.1107/S160053681203423X]

N-(2-Methylphenyl)-2-nitrobenzenesulfonamide

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S1. Comment

As part of studying the substituent effects on the structures and other aspects of *N*-(aryl)-amides (Alkan *et al.*, 2011; Bowes *et al.*, 2003; Gowda *et al.*, 1994; Saeed *et al.*, 2010; Shahwar *et al.*, 2012); *N*-arylsulfonamides (Chaithanya *et al.*, 2012; Gowda *et al.*, 2002) and *N*-chloroarylsulfonamides (Gowda & Shetty, 2004; Shetty & Gowda, 2004), in the present work, the crystal structure of *N*-(2-methylphenyl)-2-nitrobenzenesulfonamide has been determined (Fig. 1).

The conformation of the N—H bond in the —SO_2—NH— segment is *syn* to both the *ortho*-nitro group in the sulfonyl benzene ring and *ortho*-methyl group in the anilino ring. similar to that observed in *N*-(2-chlorophenyl)-2-nitrobenzene-sulfonamide (I) (Chaithanya *et al.*, 2012). The molecule is twisted at the S—N bond with the torsional angle of 73.90 (26)°, compared to the value of 74.97 (20)° in (I).

The dihedral angle between the sulfonyl and the anilino rings is 53.44 (14)°, compared to the value of 54.97 (11)° in (I).

The amide H-atom showed bifurcated intramolecular H-bonding with the O-atom of the *ortho*-nitro group in the sulfonyl benzene ring, generating S(7) motifs and the intermolecular H-bonding with the sulfonyl oxygen atom of the other molecule, generating C(4) motifs (Adsmond *et al.*, 2001; Allen *et al.*, 1998; Bernstein *et al.*, 1995; Etter, 1990).

In the crystal, the intermolecular N–H···O (S) hydrogen bonds (Table 1) link the molecules into zigzag chains. Part of the crystal structure is shown in Fig. 2.

S2. Experimental

The title compound was prepared by treating 2-nitrobenzenesulfonylchloride with 2-methylaniline in the stoichiometric ratio and boiling the reaction mixture for 15 minutes. The reaction mixture was then cooled to room temperature and added to ice cold water (100 ml). The resultant solid *N*-(2-methylphenyl)-2-nitrobenzenesulfonamide was filtered under suction and washed thoroughly with cold water and dilute HCl to remove the excess sulfonylchloride and aniline, respectively. It was then recrystallized to constant melting point (138° C) from dilute ethanol. The purity of the compound was checked and characterized by its infrared spectra.

Prism like colourless single crystals of the title compound used in X-ray diffraction studies were grown in ethanolic solution by slow evaporation of the solvent at room temperature.

S3. Refinement

H atoms bonded to C were positioned with idealized geometry using a riding model with $C_{aromatic}$—H = 0.93 Å, C_{methyl}—H = 0.96 Å. The coordinates of the amino H atom were refined with the N—H distance restrained to 0.86 (2) Å. All H atoms were refined with isotropic displacement parameters set at 1.2 $U_{eq}(C_{aromatic}, N)$ and 1.5 U_{eq} (C_{methyl}) of the parent atom.

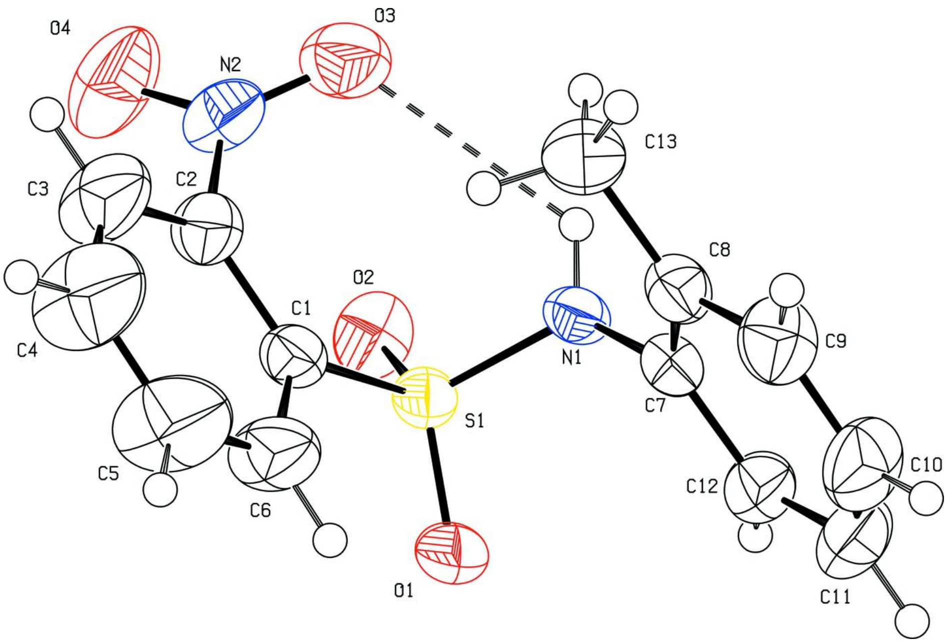

Figure 1

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

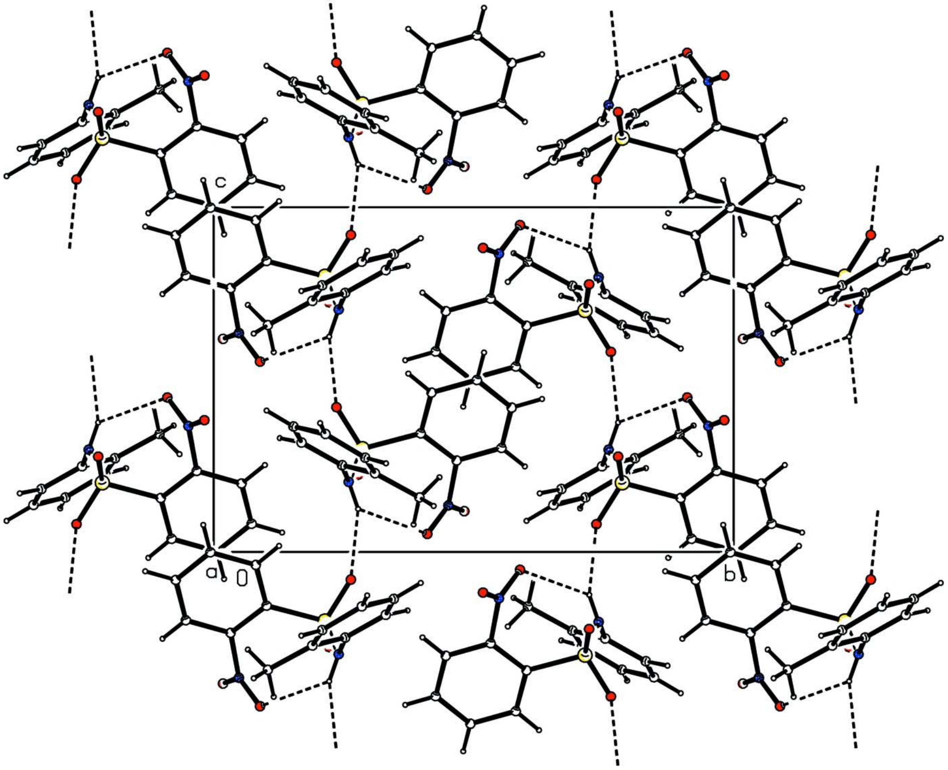

Figure 2

Molecular packing of the title compound with hydrogen bonding shown as dashed lines.

N-(2-Methylphenyl)-2-nitrobenzenesulfonamide

Crystal data

$C_{13}H_{12}N_2O_4S$
$M_r = 292.31$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.2409$ (7) Å
$b = 15.1531$ (9) Å
$c = 10.5376$ (8) Å
$\beta = 107.775$ (8)°
$V = 1405.13$ (17) Å^3
$Z = 4$

$F(000) = 608$
$D_x = 1.382$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2693 reflections
$\theta = 2.4$–27.9°
$\mu = 0.24$ mm^{-1}
$T = 293$ K
Prism, colourless
0.30 × 0.28 × 0.14 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)
$T_{\min} = 0.930$, $T_{\max} = 0.967$

5234 measured reflections
2538 independent reflections
2020 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.020$
$\theta_{\max} = 25.3°$, $\theta_{\min} = 2.4°$
$h = -11 \rightarrow 10$
$k = -13 \rightarrow 18$
$l = -12 \rightarrow 7$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.114$
$S = 1.21$
2538 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
$w = 1/[\sigma^2(F_o^2) + (0.0183P)^2 + 1.6828P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.33$ e Å^{-3}
$\Delta\rho_{\min} = -0.36$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0218 (11)

Special details

Experimental. Absorption correction: 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	$U_{\text{iso}}*/U_{\text{eq}}$
C1	0.8680 (4)	0.8982 (2)	0.1561 (3)	0.0402 (7)

C2	0.9459 (4)	0.9674 (2)	0.2349 (3)	0.0469 (8)
C3	0.9184 (5)	1.0536 (3)	0.1983 (4)	0.0751 (12)
H3	0.9734	1.0984	0.2519	0.090*
C4	0.8083 (7)	1.0730 (3)	0.0814 (5)	0.1027 (18)
H4	0.7866	1.1315	0.0561	0.123*
C5	0.7297 (6)	1.0062 (3)	0.0013 (5)	0.0987 (17)
H5	0.6558	1.0200	−0.0784	0.118*
C6	0.7592 (5)	0.9189 (3)	0.0379 (3)	0.0643 (11)
H6	0.7058	0.8743	−0.0172	0.077*
C7	0.6429 (3)	0.7470 (2)	0.2485 (3)	0.0355 (7)
C8	0.5448 (3)	0.8102 (2)	0.2718 (3)	0.0412 (7)
C9	0.3903 (4)	0.7914 (3)	0.2266 (4)	0.0575 (9)
H9	0.3219	0.8314	0.2430	0.069*
C10	0.3359 (4)	0.7149 (3)	0.1580 (4)	0.0670 (11)
H10	0.2318	0.7048	0.1266	0.080*
C11	0.4352 (4)	0.6535 (3)	0.1360 (4)	0.0617 (10)
H11	0.3984	0.6018	0.0899	0.074*
C12	0.5896 (4)	0.6689 (2)	0.1826 (3)	0.0478 (8)
H12	0.6575	0.6271	0.1699	0.057*
C13	0.5994 (4)	0.8958 (2)	0.3419 (3)	0.0534 (9)
H13A	0.6317	0.9343	0.2835	0.080*
H13B	0.6832	0.8846	0.4204	0.080*
H13C	0.5183	0.9231	0.3667	0.080*
N1	0.8048 (3)	0.75960 (17)	0.2967 (2)	0.0361 (6)
H1N	0.842 (3)	0.777 (2)	0.376 (2)	0.043*
N2	1.0601 (3)	0.95139 (19)	0.3651 (3)	0.0525 (7)
O1	0.8457 (3)	0.73616 (14)	0.0804 (2)	0.0524 (6)
O2	1.0594 (2)	0.77794 (15)	0.2763 (2)	0.0525 (6)
O3	1.0199 (3)	0.91081 (18)	0.4480 (2)	0.0641 (7)
O4	1.1859 (3)	0.9820 (2)	0.3820 (3)	0.0883 (10)
S1	0.90370 (9)	0.78584 (5)	0.20049 (7)	0.0364 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0524 (19)	0.0362 (18)	0.0334 (16)	0.0002 (14)	0.0152 (14)	0.0012 (13)
C2	0.057 (2)	0.0376 (19)	0.0454 (19)	−0.0006 (16)	0.0145 (17)	0.0021 (15)
C3	0.101 (3)	0.039 (2)	0.072 (3)	−0.005 (2)	0.007 (2)	0.005 (2)
C4	0.151 (5)	0.044 (3)	0.088 (4)	0.010 (3)	−0.001 (3)	0.021 (3)
C5	0.134 (5)	0.070 (3)	0.061 (3)	0.013 (3)	−0.017 (3)	0.019 (2)
C6	0.085 (3)	0.054 (2)	0.042 (2)	0.000 (2)	0.002 (2)	0.0026 (18)
C7	0.0391 (16)	0.0386 (17)	0.0306 (14)	0.0002 (13)	0.0132 (13)	0.0026 (13)
C8	0.0439 (18)	0.0427 (18)	0.0410 (17)	0.0008 (14)	0.0188 (15)	0.0015 (14)
C9	0.045 (2)	0.062 (2)	0.070 (2)	0.0026 (18)	0.0233 (18)	0.000 (2)
C10	0.043 (2)	0.075 (3)	0.084 (3)	−0.014 (2)	0.020 (2)	−0.007 (2)
C11	0.060 (2)	0.053 (2)	0.071 (3)	−0.0206 (19)	0.019 (2)	−0.014 (2)
C12	0.053 (2)	0.042 (2)	0.051 (2)	−0.0049 (16)	0.0201 (17)	−0.0056 (16)
C13	0.058 (2)	0.048 (2)	0.059 (2)	0.0070 (17)	0.0241 (18)	−0.0073 (17)

N1	0.0383 (14)	0.0438 (15)	0.0264 (12)	−0.0010 (11)	0.0099 (11)	−0.0014 (11)
N2	0.0560 (19)	0.0401 (16)	0.0549 (18)	−0.0031 (14)	0.0073 (15)	−0.0079 (14)
O1	0.0771 (16)	0.0457 (14)	0.0438 (12)	−0.0102 (12)	0.0323 (12)	−0.0128 (10)
O2	0.0374 (12)	0.0496 (14)	0.0725 (16)	0.0072 (10)	0.0195 (11)	−0.0014 (12)
O3	0.0784 (18)	0.0623 (17)	0.0461 (14)	−0.0001 (14)	0.0108 (13)	−0.0009 (13)
O4	0.0593 (18)	0.080 (2)	0.109 (2)	−0.0248 (16)	0.0016 (17)	−0.0003 (18)
S1	0.0422 (4)	0.0335 (4)	0.0377 (4)	0.0002 (3)	0.0183 (3)	−0.0046 (3)

Geometric parameters (Å, º)

C1—C6	1.376 (4)	C9—C10	1.376 (5)
C1—C2	1.393 (4)	C9—H9	0.9300
C1—S1	1.770 (3)	C10—C11	1.375 (5)
C2—C3	1.363 (5)	C10—H10	0.9300
C2—N2	1.473 (4)	C11—C12	1.380 (5)
C3—C4	1.369 (6)	C11—H11	0.9300
C3—H3	0.9300	C12—H12	0.9300
C4—C5	1.374 (6)	C13—H13A	0.9600
C4—H4	0.9300	C13—H13B	0.9600
C5—C6	1.382 (6)	C13—H13C	0.9600
C5—H5	0.9300	N1—S1	1.609 (2)
C6—H6	0.9300	N1—H1N	0.841 (17)
C7—C12	1.384 (4)	N2—O4	1.213 (4)
C7—C8	1.391 (4)	N2—O3	1.215 (4)
C7—N1	1.438 (4)	O1—S1	1.429 (2)
C8—C9	1.390 (4)	O2—S1	1.422 (2)
C8—C13	1.501 (4)		
C6—C1—C2	118.0 (3)	C11—C10—C9	120.2 (3)
C6—C1—S1	119.0 (3)	C11—C10—H10	119.9
C2—C1—S1	123.1 (2)	C9—C10—H10	119.9
C3—C2—C1	122.3 (3)	C10—C11—C12	119.7 (3)
C3—C2—N2	116.1 (3)	C10—C11—H11	120.1
C1—C2—N2	121.6 (3)	C12—C11—H11	120.1
C2—C3—C4	118.9 (4)	C11—C12—C7	119.6 (3)
C2—C3—H3	120.5	C11—C12—H12	120.2
C4—C3—H3	120.5	C7—C12—H12	120.2
C3—C4—C5	120.2 (4)	C8—C13—H13A	109.5
C3—C4—H4	119.9	C8—C13—H13B	109.5
C5—C4—H4	119.9	H13A—C13—H13B	109.5
C4—C5—C6	120.7 (4)	C8—C13—H13C	109.5
C4—C5—H5	119.7	H13A—C13—H13C	109.5
C6—C5—H5	119.7	H13B—C13—H13C	109.5
C1—C6—C5	119.9 (4)	C7—N1—S1	122.66 (19)
C1—C6—H6	120.0	C7—N1—H1N	117 (2)
C5—C6—H6	120.0	S1—N1—H1N	114 (2)
C12—C7—C8	121.7 (3)	O4—N2—O3	125.3 (3)
C12—C7—N1	117.6 (3)	O4—N2—C2	117.1 (3)

C8—C7—N1	120.7 (3)	O3—N2—C2	117.6 (3)
C9—C8—C7	117.1 (3)	O2—S1—O1	119.68 (14)
C9—C8—C13	120.1 (3)	O2—S1—N1	107.28 (13)
C7—C8—C13	122.8 (3)	O1—S1—N1	107.24 (13)
C10—C9—C8	121.6 (3)	O2—S1—C1	107.74 (15)
C10—C9—H9	119.2	O1—S1—C1	106.47 (14)
C8—C9—H9	119.2	N1—S1—C1	107.96 (14)

C6—C1—C2—C3	−0.3 (5)	C10—C11—C12—C7	−1.5 (5)
S1—C1—C2—C3	179.1 (3)	C8—C7—C12—C11	1.4 (5)
C6—C1—C2—N2	177.8 (3)	N1—C7—C12—C11	179.2 (3)
S1—C1—C2—N2	−2.8 (4)	C12—C7—N1—S1	76.8 (3)
C1—C2—C3—C4	1.3 (7)	C8—C7—N1—S1	−105.4 (3)
N2—C2—C3—C4	−176.9 (4)	C3—C2—N2—O4	−56.9 (5)
C2—C3—C4—C5	−1.4 (8)	C1—C2—N2—O4	125.0 (4)
C3—C4—C5—C6	0.7 (9)	C3—C2—N2—O3	121.2 (4)
C2—C1—C6—C5	−0.5 (6)	C1—C2—N2—O3	−57.0 (4)
S1—C1—C6—C5	−179.9 (4)	C7—N1—S1—O2	−170.2 (2)
C4—C5—C6—C1	0.3 (8)	C7—N1—S1—O1	−40.5 (3)
C12—C7—C8—C9	0.3 (4)	C7—N1—S1—C1	73.9 (3)
N1—C7—C8—C9	−177.4 (3)	C6—C1—S1—O2	149.0 (3)
C12—C7—C8—C13	−179.5 (3)	C2—C1—S1—O2	−30.5 (3)
N1—C7—C8—C13	2.7 (4)	C6—C1—S1—O1	19.4 (3)
C7—C8—C9—C10	−2.0 (5)	C2—C1—S1—O1	−160.0 (3)
C13—C8—C9—C10	177.8 (3)	C6—C1—S1—N1	−95.5 (3)
C8—C9—C10—C11	1.9 (6)	C2—C1—S1—N1	85.1 (3)
C9—C10—C11—C12	−0.1 (6)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.84 (2)	2.16 (2)	2.897 (3)	146 (3)
N1—H1*N*···O3	0.84 (2)	2.57 (3)	3.132 (4)	125 (3)

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