Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-(2-Methylphenyl)-4-nitrobenzenesulfonamide

U. Chaithanya,[a] Sabine Foro[b] and B. Thimme Gowda[a]*

[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

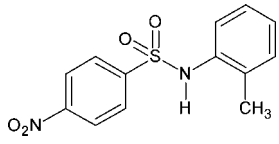
Received 19 August 2012; accepted 20 August 2012

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.046; wR factor = 0.119; data-to-parameter ratio = 15.0.

In the title compound, $C_{13}H_{12}N_2O_4S$, the dihedral angle between the planes of the rings is 51.11 (10)°. In the crystal, molecules are linked into inversion dimers through pairs of N—H···O(S) hydrogen bonds.

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.* (2005) and of *N*-chloroarylsulfonamides, see: Gowda & Shetty (2004); Shetty & Gowda (2004).



Experimental

Crystal data

$C_{13}H_{12}N_2O_4S$
M_r = 292.31
Monoclinic, $P2_1/n$
a = 14.106 (1) Å
b = 7.0082 (5) Å
c = 14.854 (2) Å
β = 110.84 (1)°
V = 1372.4 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 293 K
0.44 × 0.44 × 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} = 0.898, T_{max} = 0.942
4889 measured reflections
2781 independent reflections
2198 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.119
S = 1.08
2781 reflections
185 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O2^{i}	0.83 (2)	2.11 (2)	2.923 (2)	166 (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*.

UC thanks Mangalore University for the award of a research fellowship. BTG thanks the University Grants Commission, Government of India, New Delhi, for a special grant under the UGC–BSR one-time grant to faculty.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT6824).

References

Alkan, C., Tek, Y. & Kahraman, D. (2011). *Turk. J. Chem.* **35**, 769–777.
Bowes, K. F., Glidewell, C., Low, J. N., Skakle, J. M. S. & Wardell, J. L. (2003). *Acta Cryst.* C**59**, o1–o3.
Chaithanya, U., Foro, S. & Gowda, B. T. (2012). *Acta Cryst.* E**68**, o2745.
Gowda, B. T. & Shetty, M. (2004). *J. Phys. Org. Chem.* **17**, 848–864.
Gowda, B. T., Shetty, M. & Jayalakshmi, K. L. (2005). *Z. Naturforsch. Teil A*, **60**, 106–112.
Gowda, B. T. & Weiss, A. (1994). *Z. Naturforsch. Teil A*, **49**, 695–702.
Oxford Diffraction (2009). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Yarnton, England.
Saeed, A., Arshad, M. & Simpson, J. (2010). *Acta Cryst.* E**66**, o2808–o2809.
Shahwar, D., Tahir, M. N., Chohan, M. M., Ahmad, N. & Raza, M. A. (2012). *Acta Cryst.* E**68**, o1160.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Shetty, M. & Gowda, B. T. (2004). *Z. Naturforsch. Teil B*, **59**, 63–72.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supporting information

Acta Cryst. (2012). E**68**, o2766 [doi:10.1107/S1600536812036331]

N-(2-Methylphenyl)-4-nitrobenzenesulfonamide

U. Chaithanya, Sabine Foro and B. Thimme Gowda

S1. Comment

As a part of our studies on the substituent effects on the structures and other aspects of *N*-(aryl)-amides (Alkan *et al.*, 2011; Bowes *et al.*, 2003; Gowda & Weiss, 1994; Saeed *et al.*, 2010; Shahwar *et al.*, 2012); *N*-arylsulfonamides (Chaithanya *et al.*, 2012; Gowda *et al.*, 2005) and *N*-chloroarylsulfonamides (Gowda & Shetty, 2004; Shetty & Gowda, 2004), in the present work, the crystal structure of *N*-(2-methylphenyl)-4-nitrobenzenesulfonamide has been determined (Fig. 1).

The conformation of the N—C bond in the —SO_2—NH—C segment has *gauche* torsion with respect to the S═O bonds (Fig.1). Further, the conformation of the N—H bond in the —SO_2—NH— segment is *syn* to the *ortho*-methyl group in the anilino ring, compared to *syn* conformation observed between the N—H bond and *ortho*- nitro group in the sulfonyl benzene ring in *N*-(4-methylphenyl)-2-nitrobenzenesulfonamide (I) (Chaithanya *et al.*, 2012). The molecule is twisted at the S—N bond with the torsional angle of -58.97 (21)°, compared to the value of 76.55 (18)° in (I).

The dihedral angle between the sulfonyl and the anilino rings is 51.11 (10)°, compared to the value of 72.64 (8)° in (I).

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

S2. Experimental

The title compound was prepared by treating 4-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)-4-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 (429 K) from dilute ethanol. The purity of the compound was checked and characterized by its infrared spectra.

Prism like light brown 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 the aromatic C—H = 0.93 Å, methyl C—H = 0.96 Å. The coordinates of the amino H atom were freely 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) or 1.5 U_{eq}(C-methyl) of the parent atom.

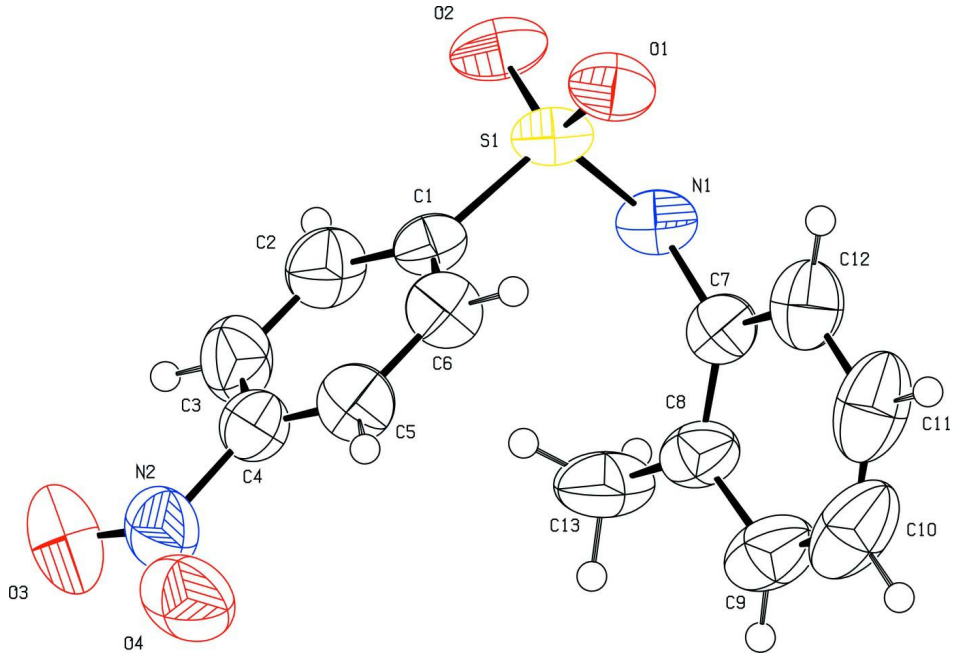


Figure 1

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

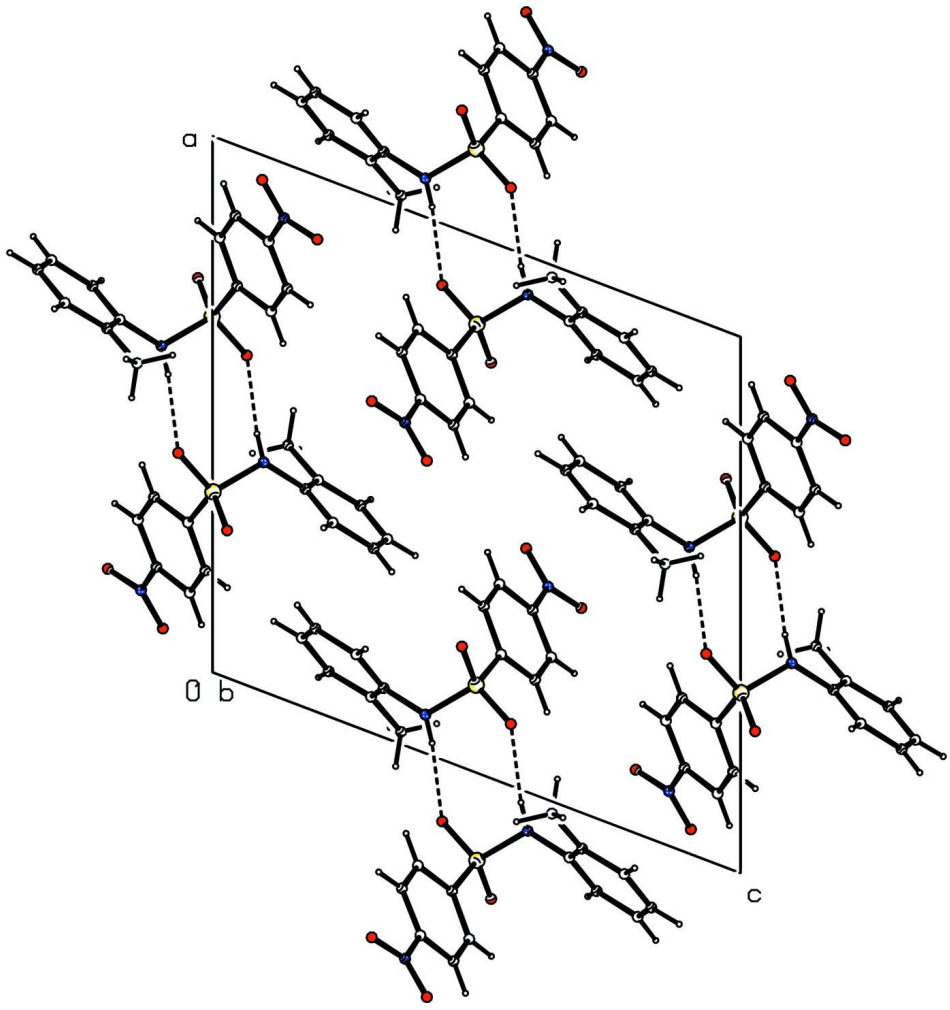


Figure 2

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

N-(2-Methylphenyl)-4-nitrobenzenesulfonamide

Crystal data

$C_{13}H_{12}N_2O_4S$
$M_r = 292.31$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 14.106$ (1) Å
$b = 7.0082$ (5) Å
$c = 14.854$ (2) Å
$\beta = 110.84$ (1)°
$V = 1372.4$ (2) Å^3
$Z = 4$
$F(000) = 608$
$D_x = 1.415$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2269 reflections
$\theta = 2.6$–27.7°
$\mu = 0.25$ mm^{-1}
$T = 293$ K
Prism, light brown
0.44 × 0.44 × 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} = 0.898, T_{max} = 0.942
4889 measured reflections
2781 independent reflections
2198 reflections with $I > 2\sigma(I)$

R_{int} = 0.016
θ_{max} = 26.4°, θ_{min} = 3.1°
h = −16→17
k = −8→8
l = −11→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.119
S = 1.08
2781 reflections
185 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
$w = 1/[\sigma^2(F_o^2) + (0.0444P)^2 + 0.7236P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
S1	0.16315 (4)	0.55405 (8)	0.49925 (4)	0.05191 (19)
O1	0.22495 (12)	0.6861 (2)	0.47241 (13)	0.0623 (4)
O2	0.11524 (13)	0.6116 (2)	0.56500 (13)	0.0680 (5)
O3	0.3815 (2)	−0.2517 (4)	0.69841 (19)	0.1191 (9)
O4	0.45351 (17)	−0.2122 (3)	0.59408 (18)	0.0960 (7)
N1	0.07213 (13)	0.4839 (3)	0.40317 (15)	0.0539 (5)
H1N	0.0239 (16)	0.439 (3)	0.4158 (18)	0.065*
N2	0.3977 (2)	−0.1605 (4)	0.63546 (19)	0.0804 (7)
C1	0.23798 (16)	0.3504 (3)	0.54651 (16)	0.0493 (5)
C2	0.2147 (2)	0.2341 (4)	0.61097 (19)	0.0690 (7)
H2	0.1633	0.2678	0.6334	0.083*
C3	0.2686 (2)	0.0682 (4)	0.6414 (2)	0.0757 (8)
H3	0.2546	−0.0116	0.6852	0.091*
C4	0.34313 (18)	0.0224 (4)	0.60623 (17)	0.0613 (6)
C5	0.36773 (18)	0.1362 (4)	0.54339 (19)	0.0642 (6)
H5	0.4190	0.1012	0.5210	0.077*

C6	0.31531 (17)	0.3033 (4)	0.51386 (18)	0.0595 (6)
H6	0.3317	0.3845	0.4721	0.071*
C7	0.09202 (15)	0.3986 (3)	0.32411 (16)	0.0516 (5)
C8	0.06673 (17)	0.2084 (4)	0.30017 (18)	0.0605 (6)
C9	0.0841 (2)	0.1372 (5)	0.2200 (2)	0.0808 (9)
H9	0.0657	0.0121	0.2006	0.097*
C10	0.1274 (2)	0.2459 (7)	0.1691 (2)	0.0949 (11)
H10	0.1386	0.1938	0.1162	0.114*
C11	0.1544 (2)	0.4303 (6)	0.1948 (2)	0.0906 (11)
H11	0.1854	0.5027	0.1606	0.109*
C12	0.13538 (18)	0.5092 (5)	0.27233 (19)	0.0704 (7)
H12	0.1518	0.6360	0.2893	0.084*
C13	0.0228 (3)	0.0823 (4)	0.3571 (2)	0.0861 (9)
H13A	−0.0455	0.1215	0.3468	0.129*
H13B	0.0631	0.0923	0.4243	0.129*
H13C	0.0227	−0.0476	0.3365	0.129*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0489 (3)	0.0462 (3)	0.0676 (4)	−0.0081 (2)	0.0293 (3)	−0.0158 (3)
O1	0.0589 (9)	0.0503 (9)	0.0836 (12)	−0.0129 (7)	0.0325 (9)	−0.0083 (8)
O2	0.0634 (10)	0.0686 (11)	0.0845 (12)	−0.0109 (8)	0.0416 (9)	−0.0321 (9)
O3	0.140 (2)	0.1042 (19)	0.0991 (18)	0.0273 (16)	0.0256 (16)	0.0432 (15)
O4	0.0757 (13)	0.0803 (14)	0.1144 (18)	0.0190 (11)	0.0122 (13)	−0.0008 (13)
N1	0.0436 (10)	0.0542 (11)	0.0688 (12)	−0.0043 (8)	0.0263 (9)	−0.0118 (9)
N2	0.0741 (15)	0.0728 (16)	0.0712 (16)	0.0040 (13)	−0.0026 (13)	0.0051 (13)
C1	0.0481 (11)	0.0503 (12)	0.0524 (12)	−0.0083 (10)	0.0216 (10)	−0.0114 (10)
C2	0.0742 (16)	0.0780 (18)	0.0681 (16)	−0.0030 (14)	0.0418 (14)	−0.0009 (14)
C3	0.0884 (19)	0.0779 (19)	0.0641 (16)	−0.0036 (16)	0.0313 (15)	0.0141 (14)
C4	0.0569 (13)	0.0594 (14)	0.0550 (13)	−0.0014 (11)	0.0044 (11)	−0.0034 (11)
C5	0.0534 (13)	0.0703 (16)	0.0696 (16)	0.0039 (12)	0.0229 (12)	0.0001 (13)
C6	0.0533 (13)	0.0629 (14)	0.0695 (15)	−0.0008 (11)	0.0306 (12)	0.0032 (12)
C7	0.0370 (10)	0.0633 (14)	0.0532 (12)	0.0069 (10)	0.0142 (9)	−0.0045 (11)
C8	0.0497 (12)	0.0661 (15)	0.0635 (14)	0.0088 (11)	0.0174 (11)	−0.0150 (12)
C9	0.0664 (16)	0.097 (2)	0.0754 (18)	0.0128 (16)	0.0213 (15)	−0.0277 (17)
C10	0.0700 (18)	0.153 (4)	0.0624 (18)	0.019 (2)	0.0248 (15)	−0.021 (2)
C11	0.0658 (17)	0.149 (4)	0.0614 (17)	0.002 (2)	0.0279 (14)	0.012 (2)
C12	0.0561 (14)	0.0890 (19)	0.0644 (15)	0.0011 (13)	0.0193 (12)	0.0088 (14)
C13	0.108 (2)	0.0572 (16)	0.102 (2)	−0.0184 (16)	0.049 (2)	−0.0244 (16)

Geometric parameters (Å, º)

S1—O1	1.4221 (16)	C5—H5	0.9300
S1—O2	1.4292 (16)	C6—H6	0.9300
S1—N1	1.620 (2)	C7—C12	1.379 (3)
S1—C1	1.765 (2)	C7—C8	1.393 (3)
O3—N2	1.220 (3)	C8—C9	1.391 (3)

O4—N2	1.214 (3)	C8—C13	1.501 (4)
N1—C7	1.431 (3)	C9—C10	1.361 (5)
N1—H1N	0.830 (16)	C9—H9	0.9300
N2—C4	1.479 (3)	C10—C11	1.363 (5)
C1—C6	1.382 (3)	C10—H10	0.9300
C1—C2	1.383 (3)	C11—C12	1.387 (4)
C2—C3	1.375 (4)	C11—H11	0.9300
C2—H2	0.9300	C12—H12	0.9300
C3—C4	1.369 (4)	C13—H13A	0.9600
C3—H3	0.9300	C13—H13B	0.9600
C4—C5	1.363 (3)	C13—H13C	0.9600
C5—C6	1.371 (3)		

O1—S1—O2	119.61 (10)	C5—C6—C1	119.7 (2)
O1—S1—N1	108.86 (11)	C5—C6—H6	120.2
O2—S1—N1	105.57 (10)	C1—C6—H6	120.2
O1—S1—C1	107.34 (10)	C12—C7—C8	121.3 (2)
O2—S1—C1	108.52 (11)	C12—C7—N1	118.6 (2)
N1—S1—C1	106.21 (10)	C8—C7—N1	120.1 (2)
C7—N1—S1	121.63 (14)	C9—C8—C7	117.0 (3)
C7—N1—H1N	116.3 (18)	C9—C8—C13	120.3 (3)
S1—N1—H1N	111.9 (18)	C7—C8—C13	122.6 (2)
O4—N2—O3	124.3 (3)	C10—C9—C8	121.7 (3)
O4—N2—C4	118.3 (3)	C10—C9—H9	119.2
O3—N2—C4	117.3 (3)	C8—C9—H9	119.2
C6—C1—C2	120.9 (2)	C9—C10—C11	120.7 (3)
C6—C1—S1	119.24 (18)	C9—C10—H10	119.6
C2—C1—S1	119.75 (18)	C11—C10—H10	119.6
C3—C2—C1	119.2 (2)	C10—C11—C12	119.5 (3)
C3—C2—H2	120.4	C10—C11—H11	120.2
C1—C2—H2	120.4	C12—C11—H11	120.2
C4—C3—C2	118.8 (2)	C7—C12—C11	119.6 (3)
C4—C3—H3	120.6	C7—C12—H12	120.2
C2—C3—H3	120.6	C11—C12—H12	120.2
C5—C4—C3	122.8 (2)	C8—C13—H13A	109.5
C5—C4—N2	118.4 (2)	C8—C13—H13B	109.5
C3—C4—N2	118.8 (3)	H13A—C13—H13B	109.5
C4—C5—C6	118.6 (2)	C8—C13—H13C	109.5
C4—C5—H5	120.7	H13A—C13—H13C	109.5
C6—C5—H5	120.7	H13B—C13—H13C	109.5

O1—S1—N1—C7	56.3 (2)	C3—C4—C5—C6	−0.3 (4)
O2—S1—N1—C7	−174.08 (18)	N2—C4—C5—C6	178.1 (2)
C1—S1—N1—C7	−59.0 (2)	C4—C5—C6—C1	−1.3 (4)
O1—S1—C1—C6	−29.1 (2)	C2—C1—C6—C5	2.0 (4)
O2—S1—C1—C6	−159.71 (18)	S1—C1—C6—C5	−174.02 (19)
N1—S1—C1—C6	87.2 (2)	S1—N1—C7—C12	−66.4 (3)
O1—S1—C1—C2	154.79 (19)	S1—N1—C7—C8	114.3 (2)

O2—S1—C1—C2	24.2 (2)	C12—C7—C8—C9	−2.1 (3)
N1—S1—C1—C2	−88.9 (2)	N1—C7—C8—C9	177.3 (2)
C6—C1—C2—C3	−1.0 (4)	C12—C7—C8—C13	177.8 (2)
S1—C1—C2—C3	175.0 (2)	N1—C7—C8—C13	−2.9 (3)
C1—C2—C3—C4	−0.6 (4)	C7—C8—C9—C10	2.4 (4)
C2—C3—C4—C5	1.2 (4)	C13—C8—C9—C10	−177.5 (3)
C2—C3—C4—N2	−177.1 (2)	C8—C9—C10—C11	−0.7 (5)
O4—N2—C4—C5	−8.4 (4)	C9—C10—C11—C12	−1.5 (5)
O3—N2—C4—C5	173.3 (3)	C8—C7—C12—C11	0.0 (4)
O4—N2—C4—C3	170.0 (3)	N1—C7—C12—C11	−179.4 (2)
O3—N2—C4—C3	−8.2 (4)	C10—C11—C12—C7	1.9 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O2^{i}	0.83 (2)	2.11 (2)	2.923 (2)	166 (2)

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