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N-(4-Methoxybenzoyl)-2-methylbenzenesulfonamide

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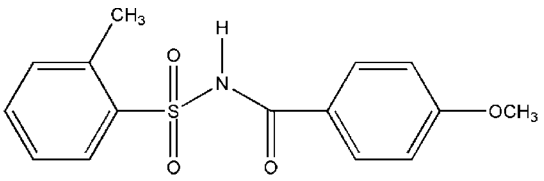
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.046; wR factor = 0.122; data-to-parameter ratio = 12.4.

In the title compound, $C_{15}H_{15}NO_4S$, the dihedral angle between the aromatic rings is 80.81 (1)° and the dihedral angle between the planes defined by the S—N—C=O fragment and the sulfonyl benzene ring is 86.34 (1)°. In the extended structure, dimers related by a crystallographic twofold axis are connected by pairs of both N—H⋯O hydrogen bonds and C—H⋯O interactions, which generate $R_2^2(8)$ and $R_2^2(14)$ loops, respectively. A weak aromatic π–π stacking interaction is also observed [centroid–centroid separation = 3.7305 (3) Å].

Related literature

For related structures, see: Gowda *et al.* (2010); Suchetan *et al.* (2010*a*,*b*, 2011); Sreenivasa *et al.* (2013, 2014).



Experimental

Crystal data

$C_{15}H_{15}NO_4S$
M_r = 305.34
Monoclinic, $C2/c$
a = 21.807 (2) Å
b = 7.3521 (8) Å
c = 18.602 (2) Å
β = 101.211 (3)°
V = 2925.4 (5) Å^3
Z = 8
Cu $K\alpha$ radiation
μ = 2.11 mm^{-1}
T = 293 K
0.38 × 0.29 × 0.22 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{\min}$ = 0.504, $T_{\max}$ = 0.629
16411 measured reflections
2431 independent reflections
2174 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.060

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.122
S = 0.92
2431 reflections
196 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ = 0.34 e Å^{-3}
$\Delta\rho_{\min}$ = −0.51 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1⋯O2$^{\rm i}$	0.81 (3)	2.16 (3)	2.917 (2)	164 (3)
C13—H13⋯O2$^{\rm i}$	0.93	2.56	3.288 (3)	136

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT-Plus* (Bruker, 2009); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97*.

The authors acknowledge the IOE X-ray diffractometer facility, University of Mysore, Mysore, for the data collection.

Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7188).

References

Bruker (2009). *APEX2, SADABS and SAINT-Plus.* Bruker AXS Inc., Madison, Wisconsin, USA.
Gowda, B. T., Foro, S., Suchetan, P. A. & Fuess, H. (2010). *Acta Cryst.* E**66**, o747.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sreenivasa, S., Nanjundaswamy, M. S., Madankumar, S., Lokanath, N. K., Suresha, E. & Suchetan, P. A. (2014). *Acta Cryst.* E**70**, o192.
Sreenivasa, S., Palakshamurthy, B. S., Tonannavar, J., Jayashree, Y., Sudha, A. G. & Suchetan, P. A. (2013). *Acta Cryst.* E**69**, o1664–o1665.
Suchetan, P. A., Foro, S. & Gowda, B. T. (2011). *Acta Cryst.* E**67**, o929.
Suchetan, P. A., Gowda, B. T., Foro, S. & Fuess, H. (2010*a*). *Acta Cryst.* E**66**, o1024.
Suchetan, P. A., Gowda, B. T., Foro, S. & Fuess, H. (2010*b*). *Acta Cryst.* E**66**, o1997.

supporting information

Acta Cryst. (2014). E**70**, o193 [doi:10.1107/S1600536814001354]

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S1. Introduction

As a part of our continued efforts to study the crystal structures of N-(aroyl)-arylsulfonamides (Sreenivasa *et al.*, 2014), we report here the crystal structure of the title compound (I) (Fig 1).

S2. Experimental

S2.1. Synthesis and crystallization

The title compound (I) was prepared by refluxing a mixture of 4-methoxybenzoic acid, 2-methylbenzenesulfonamide and phosphorous oxychloride ($POCl_3$) for 2 h on a water bath. The resultant mixture was cooled and poured into ice cold water. The solid obtained was filtered and washed thoroughly with water and then dissolved in sodium bicarbonate solution. The compound was later reprecipitated by acidifying the filtered solution with dilute HCl. The compound obtained was filtered and later dried (Melting point: 447 K).

Colorless prisms of (I) were obtained from a slow evaporation of its aqueous methanolic solution at room temperature.

S2.2. Refinement

The H atom of the NH group was located in a difference map and later refined freely. 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 1.2-1.5 times of the U eq of the parent atom).

S3. Results and discussion

In I, the dihedral angle between the two aromatic rings is 80.81 (1)°. Compared to this, the dihedral angle is 73.9 (1)° in N-(benzoyl)-2-methylbenzenesulfonamide (II, Suchetan *et al.*, 2010*a*), 89.4 (1)° and 82.4 (1)° respectively in the two molecules in the asymmetric unit of N-(4-chlorobenzoyl)-2-methylbenzenesulfonamide (III, Suchetan *et al.*, 2010*b*), 88.1 (1)° and 83.5 (1)° respectively in the two molecules in the asymmetric unit of N-(4-methylbenzoyl)-2-methylbenzenesulfonamide (IV, Gowda *et al.*, 2010) and 83.8 (2)° in N-(4-nitrobenzoyl)-2-methylbenzenesulfonamide (V, Suchetan *et al.*, 2011). This shows that introducing a substituent into the para position of the benzoyl ring of II correlates with a increase of the dihedral angle between the aromatic rings. In contrast to this, the dihedral angle is small in N-(4-methoxy-benzoyl)-benzenesulfonamide (VI, Sreenivasa *et al.*, 2014) and N-(4-methoxybenzoyl)-4-methylbenzene-sulfonamide (VII, Sreenivasa *et al.*, 2013), the dihedral angle being respectively 69.81 (1)° and 78.62 (16)° in VI and VII. Further, the molecule is twisted at the S atom, the dihedral angle between the planes defined by the S—N—C=O segment in the central chain and the sulfonyl benzene ring being 86.34 (1)°.

The supramolecular architecture of I is built in three stages. In the first stage, the molecules are linked into dimers by a crystallographic twofold axis through strong N1—H1···O2 hydrogen bonds, thus generating $R_2^2(8)$ rings (Figure 2). These dimers in the second stage are linked through an additional C13—H13···O2 interaction (Figure 2) forming $R_2^2(14)$ ring motif. In the third stage, π(methylphenyl) $\cdots\pi$(methylphenyl) interactions stabilize the structure, Cg(methylphenyl) $\cdots$Cg(methylphenyl) distance being 3.7305 (3)Å (Figure 3). The geometries and symmetry operations of various interactions are shown in Table 1.

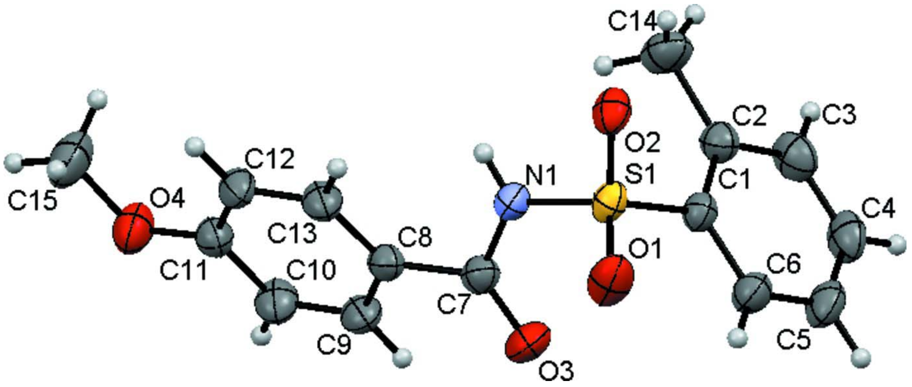

Figure 1

Molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.

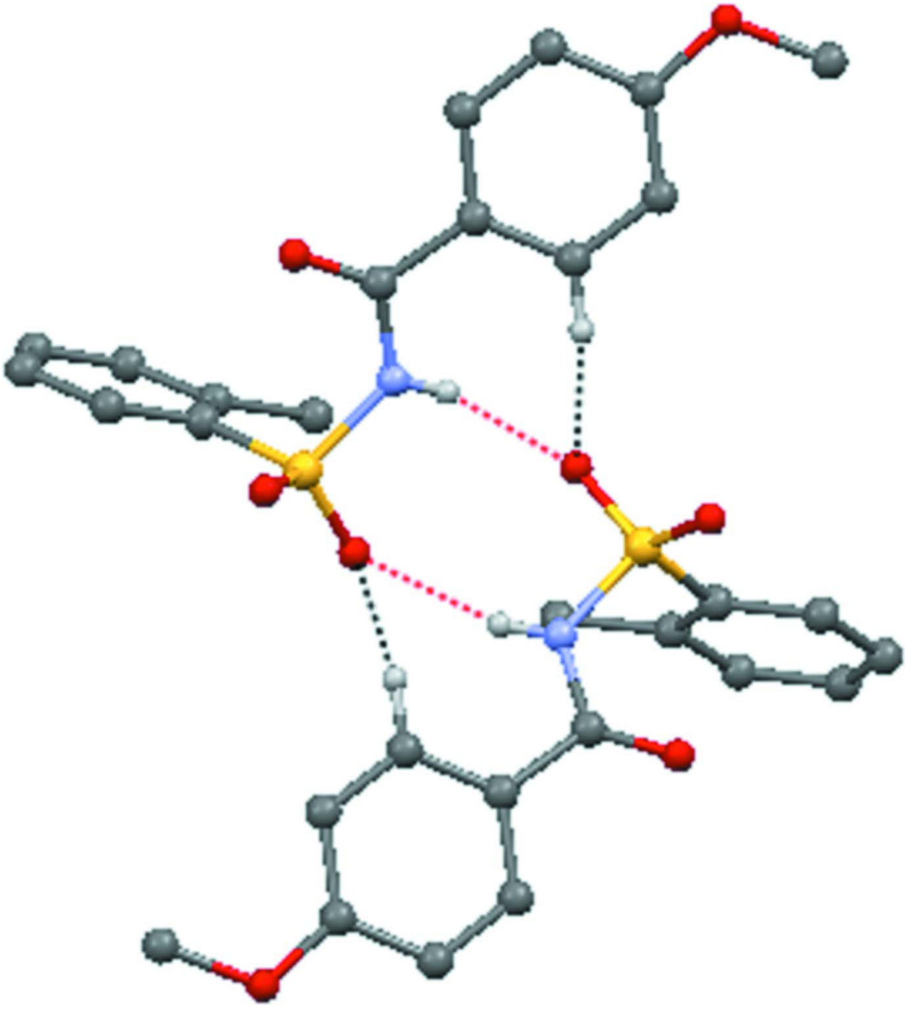

Figure 2

Formation of $R_2^2(8)$ and $R_2^2(14)$ rings in I.

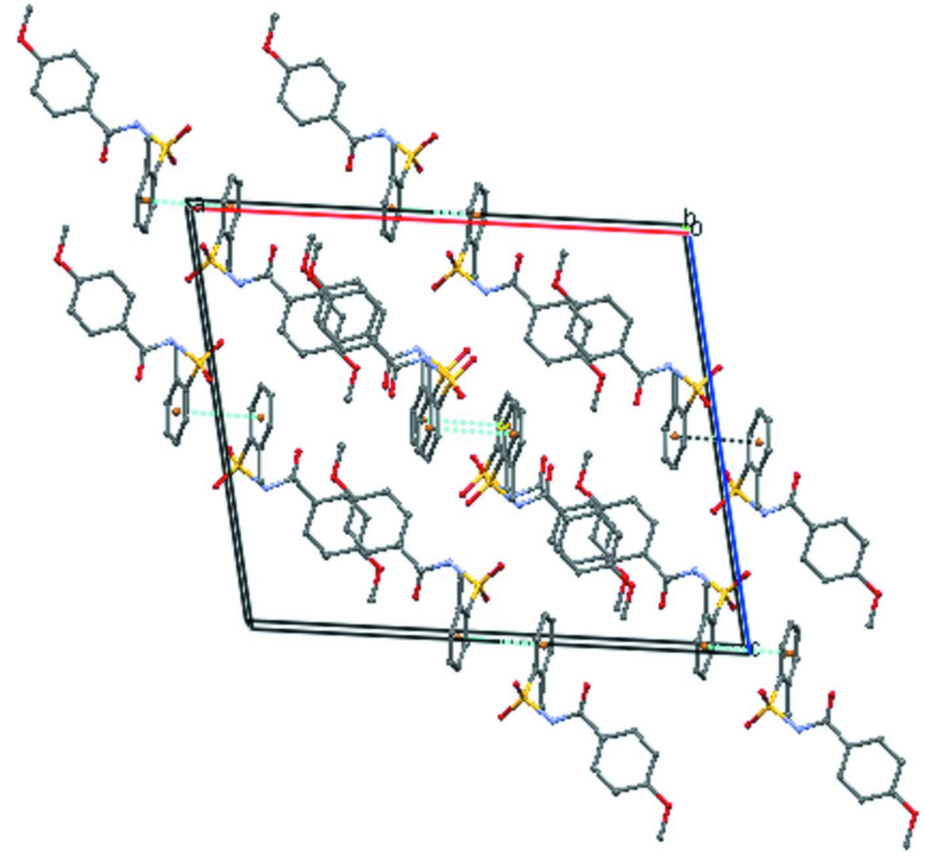

Figure 3

$\pi\cdots\pi$ interaction observed in the crystal structure. *Cg* is the centroid of the methylphenyl ring.

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Crystal data

$C_{15}H_{15}NO_4S$
M_r = 305.34
Monoclinic, $C2/c$
Hall symbol: -C 2yc
a = 21.807 (2) Å
b = 7.3521 (8) Å
c = 18.602 (2) Å
β = 101.211 (3)°
V = 2925.4 (5) Å^3
Z = 8
F(000) = 1280

Prism
D_x = 1.387 Mg m^{-3}
Melting point: 447 K
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 25 reflections
θ = 4.1–64.7°
μ = 2.11 mm^{-1}
T = 293 K
Prism, colourless
0.38 × 0.29 × 0.22 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator

phi and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{\min}$ = 0.504, $T_{\max}$ = 0.629

16411 measured reflections
2431 independent reflections
2174 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.060$

$\theta_{\max} = 64.7°$, $\theta_{\min} = 4.1°$
$h = -25 \rightarrow 24$
$k = -7 \rightarrow 8$
$l = -20 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.122$
$S = 0.92$
2431 reflections
196 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0923P)^2 + 2.3453P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.34$ e Å^{-3}
$\Delta\rho_{\min} = -0.51$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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}
H1	0.0708 (12)	0.197 (3)	0.2813 (15)	0.051 (7)*
S1	0.02647 (2)	0.20056 (7)	0.37394 (2)	0.0326 (2)
O2	−0.02356 (6)	0.2872 (2)	0.32370 (7)	0.0408 (4)
O1	0.01308 (8)	0.0360 (2)	0.40868 (8)	0.0499 (4)
O3	0.15501 (7)	0.0483 (2)	0.41305 (7)	0.0464 (4)
C8	0.17364 (9)	0.0372 (3)	0.29109 (9)	0.0335 (4)
N1	0.07872 (7)	0.1589 (2)	0.32300 (8)	0.0346 (4)
O4	0.28653 (7)	−0.0475 (2)	0.13921 (8)	0.0512 (4)
C13	0.14633 (9)	0.0201 (3)	0.21740 (10)	0.0395 (5)
H13	0.1031	0.0286	0.2031	0.047*
C6	0.07042 (10)	0.2955 (3)	0.51353 (10)	0.0406 (5)
H6	0.0606	0.1764	0.5238	0.049*
C12	0.18241 (10)	−0.0093 (3)	0.16511 (10)	0.0399 (5)
H12	0.1636	−0.0204	0.1160	0.048*
C2	0.07397 (9)	0.5358 (3)	0.42381 (10)	0.0372 (5)
C1	0.06024 (8)	0.3584 (3)	0.44137 (9)	0.0317 (4)
C7	0.13675 (9)	0.0789 (3)	0.34846 (9)	0.0341 (4)
C11	0.24672 (10)	−0.0224 (3)	0.18620 (10)	0.0372 (5)
C10	0.27437 (10)	−0.0116 (3)	0.26017 (10)	0.0439 (5)
H10	0.3175	−0.0233	0.2746	0.053*

C3	0.09891 (10)	0.6503 (3)	0.48231 (12)	0.0476 (5)
H3	0.1087	0.7699	0.4729	0.057*
C4	0.10915 (10)	0.5888 (3)	0.55374 (11)	0.0488 (6)
H4	0.1257	0.6677	0.5917	0.059*
C9	0.23797 (10)	0.0164 (3)	0.31184 (10)	0.0402 (5)
H9	0.2566	0.0214	0.3612	0.048*
C5	0.09543 (11)	0.4140 (4)	0.56954 (10)	0.0484 (6)
H5	0.1029	0.3744	0.6179	0.058*
C14	0.06366 (13)	0.6127 (3)	0.34685 (11)	0.0543 (6)
H14A	0.0891	0.5479	0.3188	0.082*
H14B	0.0749	0.7392	0.3489	0.082*
H14C	0.0204	0.5999	0.3240	0.082*
C15	0.25999 (13)	−0.0489 (5)	0.06265 (12)	0.0653 (8)
H15A	0.2278	−0.1398	0.0529	0.098*
H15B	0.2920	−0.0762	0.0354	0.098*
H15C	0.2423	0.0682	0.0483	0.098*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0365 (3)	0.0348 (3)	0.0268 (3)	−0.00716 (18)	0.00702 (19)	−0.00524 (15)
O2	0.0324 (7)	0.0554 (10)	0.0325 (6)	0.0007 (6)	0.0010 (5)	−0.0104 (6)
O1	0.0681 (10)	0.0406 (10)	0.0444 (8)	−0.0208 (8)	0.0191 (7)	−0.0039 (6)
O3	0.0543 (9)	0.0550 (10)	0.0273 (7)	0.0034 (7)	0.0017 (6)	0.0049 (6)
C8	0.0386 (10)	0.0296 (11)	0.0303 (9)	0.0028 (8)	0.0019 (7)	−0.0019 (7)
N1	0.0392 (9)	0.0399 (10)	0.0253 (7)	0.0049 (7)	0.0074 (6)	0.0007 (6)
O4	0.0439 (8)	0.0725 (12)	0.0379 (7)	0.0054 (8)	0.0095 (6)	−0.0081 (7)
C13	0.0351 (10)	0.0450 (13)	0.0345 (10)	0.0073 (9)	−0.0032 (8)	−0.0057 (8)
C6	0.0489 (11)	0.0422 (13)	0.0307 (9)	−0.0017 (9)	0.0075 (8)	−0.0004 (8)
C12	0.0430 (11)	0.0445 (12)	0.0287 (9)	0.0080 (9)	−0.0013 (7)	−0.0074 (8)
C2	0.0403 (10)	0.0341 (12)	0.0357 (9)	0.0005 (9)	0.0034 (7)	−0.0032 (8)
C1	0.0326 (9)	0.0352 (11)	0.0266 (8)	−0.0011 (8)	0.0046 (6)	−0.0040 (7)
C7	0.0403 (10)	0.0304 (11)	0.0302 (9)	−0.0022 (8)	0.0035 (7)	−0.0012 (7)
C11	0.0396 (10)	0.0358 (12)	0.0356 (9)	0.0046 (8)	0.0059 (8)	−0.0039 (7)
C10	0.0332 (10)	0.0562 (15)	0.0388 (10)	0.0049 (9)	−0.0020 (8)	−0.0069 (9)
C3	0.0514 (12)	0.0351 (12)	0.0534 (12)	−0.0048 (10)	0.0030 (9)	−0.0116 (9)
C4	0.0499 (12)	0.0511 (15)	0.0420 (11)	−0.0006 (11)	0.0004 (9)	−0.0205 (9)
C9	0.0414 (11)	0.0449 (13)	0.0301 (9)	0.0061 (9)	−0.0039 (7)	−0.0031 (8)
C5	0.0545 (12)	0.0619 (16)	0.0267 (9)	0.0014 (11)	0.0027 (8)	−0.0083 (9)
C14	0.0776 (16)	0.0396 (14)	0.0432 (11)	−0.0074 (12)	0.0051 (10)	0.0074 (9)
C15	0.0629 (15)	0.099 (2)	0.0353 (11)	0.0046 (15)	0.0129 (10)	−0.0059 (11)

Geometric parameters (Å, º)

S1—O1	1.4283 (15)	C2—C1	1.391 (3)
S1—O2	1.4398 (14)	C2—C3	1.399 (3)
S1—N1	1.6461 (16)	C2—C14	1.515 (3)
S1—C1	1.7613 (18)	C11—C10	1.393 (3)

O3—C7	1.211 (2)	C10—C9	1.376 (3)
C8—C13	1.390 (2)	C10—H10	0.9300
C8—C9	1.389 (3)	C3—C4	1.380 (3)
C8—C7	1.488 (3)	C3—H3	0.9300
N1—C7	1.392 (2)	C4—C5	1.364 (4)
N1—H1	0.81 (3)	C4—H4	0.9300
O4—C11	1.359 (2)	C9—H9	0.9300
O4—C15	1.429 (3)	C5—H5	0.9300
C13—C12	1.382 (3)	C14—H14A	0.9600
C13—H13	0.9300	C14—H14B	0.9600
C6—C5	1.386 (3)	C14—H14C	0.9600
C6—C1	1.396 (3)	C15—H15A	0.9600
C6—H6	0.9300	C15—H15B	0.9600
C12—C11	1.384 (3)	C15—H15C	0.9600
C12—H12	0.9300		

O1—S1—O2	118.20 (10)	O4—C11—C12	124.46 (17)
O1—S1—N1	109.14 (9)	O4—C11—C10	115.78 (18)
O2—S1—N1	103.34 (8)	C12—C11—C10	119.75 (18)
O1—S1—C1	109.25 (9)	C9—C10—C11	120.05 (19)
O2—S1—C1	109.27 (9)	C9—C10—H10	120.0
N1—S1—C1	107.00 (8)	C11—C10—H10	120.0
C13—C8—C9	118.68 (18)	C4—C3—C2	121.2 (2)
C13—C8—C7	122.57 (18)	C4—C3—H3	119.4
C9—C8—C7	118.75 (16)	C2—C3—H3	119.4
C7—N1—S1	124.60 (13)	C5—C4—C3	120.98 (19)
C7—N1—H1	118.8 (19)	C5—C4—H4	119.5
S1—N1—H1	116.3 (19)	C3—C4—H4	119.5
C11—O4—C15	117.14 (17)	C10—C9—C8	120.73 (17)
C12—C13—C8	121.00 (18)	C10—C9—H9	119.6
C12—C13—H13	119.5	C8—C9—H9	119.6
C8—C13—H13	119.5	C4—C5—C6	120.05 (19)
C5—C6—C1	118.7 (2)	C4—C5—H5	120.0
C5—C6—H6	120.6	C6—C5—H5	120.0
C1—C6—H6	120.6	C2—C14—H14A	109.5
C13—C12—C11	119.69 (17)	C2—C14—H14B	109.5
C13—C12—H12	120.2	H14A—C14—H14B	109.5
C11—C12—H12	120.2	C2—C14—H14C	109.5
C1—C2—C3	116.76 (18)	H14A—C14—H14C	109.5
C1—C2—C14	124.95 (17)	H14B—C14—H14C	109.5
C3—C2—C14	118.3 (2)	O4—C15—H15A	109.5
C2—C1—C6	122.33 (17)	O4—C15—H15B	109.5
C2—C1—S1	121.98 (13)	H15A—C15—H15B	109.5
C6—C1—S1	115.67 (16)	O4—C15—H15C	109.5
O3—C7—N1	121.18 (17)	H15A—C15—H15C	109.5
O3—C7—C8	123.64 (18)	H15B—C15—H15C	109.5
N1—C7—C8	115.18 (15)		

O1—S1—N1—C7	−55.03 (19)	S1—N1—C7—C8	174.04 (14)
O2—S1—N1—C7	178.35 (16)	C13—C8—C7—O3	159.9 (2)
C1—S1—N1—C7	63.07 (19)	C9—C8—C7—O3	−20.8 (3)
C9—C8—C13—C12	−2.7 (3)	C13—C8—C7—N1	−20.8 (3)
C7—C8—C13—C12	176.6 (2)	C9—C8—C7—N1	158.54 (19)
C8—C13—C12—C11	0.1 (3)	C15—O4—C11—C12	4.1 (3)
C3—C2—C1—C6	0.1 (3)	C15—O4—C11—C10	−176.6 (2)
C14—C2—C1—C6	−179.6 (2)	C13—C12—C11—O4	−178.6 (2)
C3—C2—C1—S1	178.06 (16)	C13—C12—C11—C10	2.1 (3)
C14—C2—C1—S1	−1.6 (3)	O4—C11—C10—C9	179.1 (2)
C5—C6—C1—C2	−0.4 (3)	C12—C11—C10—C9	−1.6 (4)
C5—C6—C1—S1	−178.41 (16)	C1—C2—C3—C4	0.0 (3)
O1—S1—C1—C2	−175.20 (16)	C14—C2—C3—C4	179.7 (2)
O2—S1—C1—C2	−44.48 (18)	C2—C3—C4—C5	0.2 (4)
N1—S1—C1—C2	66.76 (18)	C11—C10—C9—C8	−1.1 (4)
O1—S1—C1—C6	2.85 (18)	C13—C8—C9—C10	3.2 (3)
O2—S1—C1—C6	133.58 (15)	C7—C8—C9—C10	−176.1 (2)
N1—S1—C1—C6	−115.18 (16)	C3—C4—C5—C6	−0.4 (3)
S1—N1—C7—O3	−6.6 (3)	C1—C6—C5—C4	0.5 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.81 (3)	2.16 (3)	2.917 (2)	164 (3)
C13—H13···O2^{i}	0.93	2.56	3.288 (3)	136

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