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## Structure Reports

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2-Chloro-*N*-(4-methoxybenzoyl)benzenesulfonamideS Sreenivasa,<sup>a</sup> B. S. Palakshamurthy,<sup>b</sup> K. J. Pampa,<sup>c</sup>  
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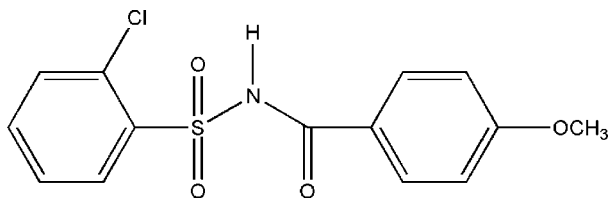
Received 19 January 2014; accepted 21 January 2014

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.063;  $wR$  factor = 0.163; data-to-parameter ratio = 12.6.

In the title compound,  $\text{C}_{14}\text{H}_{12}\text{ClNO}_4\text{S}$ , the dihedral angle between the aromatic rings is  $82.07(1)^\circ$  and the dihedral angle between the planes defined by the  $\text{S}-\text{N}-\text{C}=\text{O}$  fragment and the sulfonyl benzene ring is  $82.46(3)^\circ$ . In the crystal, the molecules are linked into  $C(4)$  chains running along  $[001]$  by strong  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds. A  $\text{C}-\text{H}\cdots\text{O}$  interaction reinforces the  $[001]$  chains: its graph-set symbol is  $C(7)$ . The chains are cross-linked into  $(100)$  sheets by further  $\text{C}-\text{H}\cdots\text{O}$  interactions as  $C(6)$  chains along  $[001]$ . The structure also features weak  $\pi-\pi$  stacking interactions [centroid-centroid distances =  $3.577(1)$  and  $3.8016(1)$  Å].

## Related literature

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



## Experimental

## Crystal data

$\text{C}_{14}\text{H}_{12}\text{ClNO}_4\text{S}$   
 $M_r = 325.76$   
Monoclinic,  $P2_1/c$   
 $a = 14.5293(19)$  Å  
 $b = 10.6225(14)$  Å  
 $c = 9.6918(14)$  Å  
 $\beta = 92.695(5)^\circ$   
 $V = 1494.2(4)$  Å<sup>3</sup>  
 $Z = 4$   
Cu  $K\alpha$  radiation  
 $\mu = 3.71$  mm<sup>-1</sup>  
 $T = 293$  K  
 $0.44 \times 0.35 \times 0.26$  mm

## Data collection

Bruker APEXII CCD diffractometer  
Absorption correction: multi-scan (SADABS; Bruker, 2009)  
 $T_{\min} = 0.263$ ,  $T_{\max} = 0.381$   
17846 measured reflections  
2449 independent reflections  
2295 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.058$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.063$   
 $wR(F^2) = 0.163$   
 $S = 1.11$   
2449 reflections  
195 parameters  
H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.52$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.37$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}\cdots\text{O2}^i$	0.81 (3)	2.09 (3)	2.872 (3)	172 (3)
$\text{C3}-\text{H3}\cdots\text{O1}^{\text{H}}$	0.93	2.57	3.370 (4)	144
$\text{C9}-\text{H9}\cdots\text{O2}^i$	0.93	2.48	3.288 (3)	145

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

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: HB7189).

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## supporting information

*Acta Cryst.* (2014). E70, o199 [doi:10.1107/S1600536814001482]

**2-Chloro-*N*-(4-methoxybenzoyl)benzenesulfonamide**

**S Sreenivasa, B. S. Palakshamurthy, K. J. Pampa, N. K. Lokanath and P. A. Suchetan**

**S1. Introduction**

As a part of our continued structural studies of *N*-(aroyl)-arylsulfonamides (Suchetan *et al.*, 2011*a*, 2011*b*; Gowda *et al.*, 2010*a*, 2010*b*), 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-chlorobenzenesulfonamide and phosphorous oxychloride (POCl<sub>3</sub>) 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: 445 K).

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

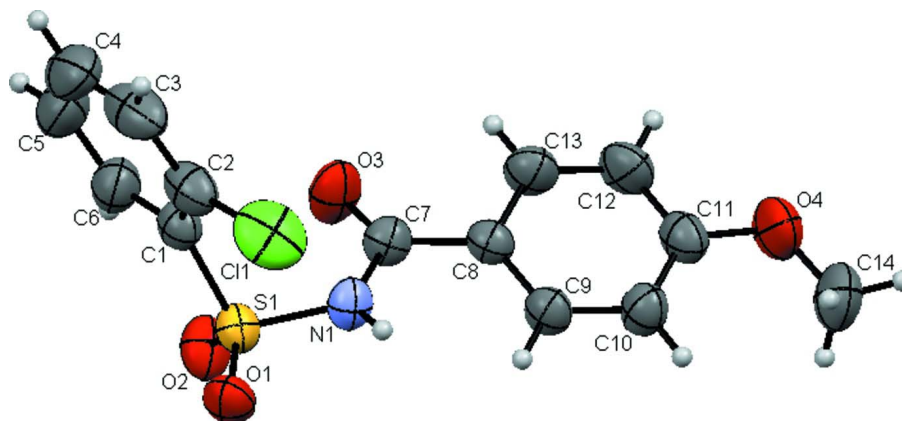
**S2.2. Refinement**

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).

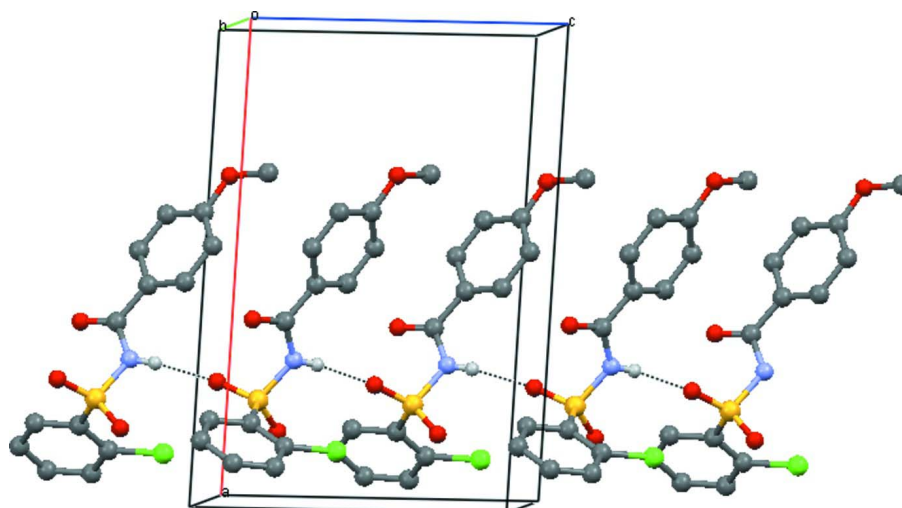
**S3. Results and discussion**

In I, the dihedral angle between the two aromatic rings is 82.07 (1)°. Compared to this, the dihedral angle is 73.3 (1)° in *N*-(benzoyl)-2-chlorobenzenesulfonamide (II, Gowda *et al.*, 2010*a*), 85.7 (1)° in *N*-(4-chlorobenzoyl)-2-chlorobenzenesulfonamide (III, Suchetan *et al.*, 2011*a*), 89.1 (2)° in *N*-(4-methylbenzoyl)-2-chlorobenzenesulfonamide (IV, Gowda *et al.*, 2010*b*) and 85.4 (1)° in *N*-(4-nitrobenzoyl)-2-chlorobenzenesulfonamide (V, Suchetan *et al.*, 2011*b*). 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. 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 82.46 (3)°.

In the crystal structure, the molecules are linked into C(4) chains running along [001] through strong N1—H1⋯O2 hydrogen bonds (Figure 2). The molecules are further linked into one another through C3—H3⋯O1 (Figure 3) and C9—H9⋯O2 (Figure 4) interactions into C(6) and C(7) chains running along [001]. The structure is further stabilized by two alternate  $\pi$ - $\pi$  interactions, Cg(methoxyphenyl)-Cg(methoxyphenyl) and Cg(chlorophenyl)-Cg(chlorophenyl) distances being respectively 3.577 (1)Å and 3.8016 (1)Å.

**Figure 1**

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

**Figure 2**

Formation of C(4) chains through N—H...O hydrogen bonds.

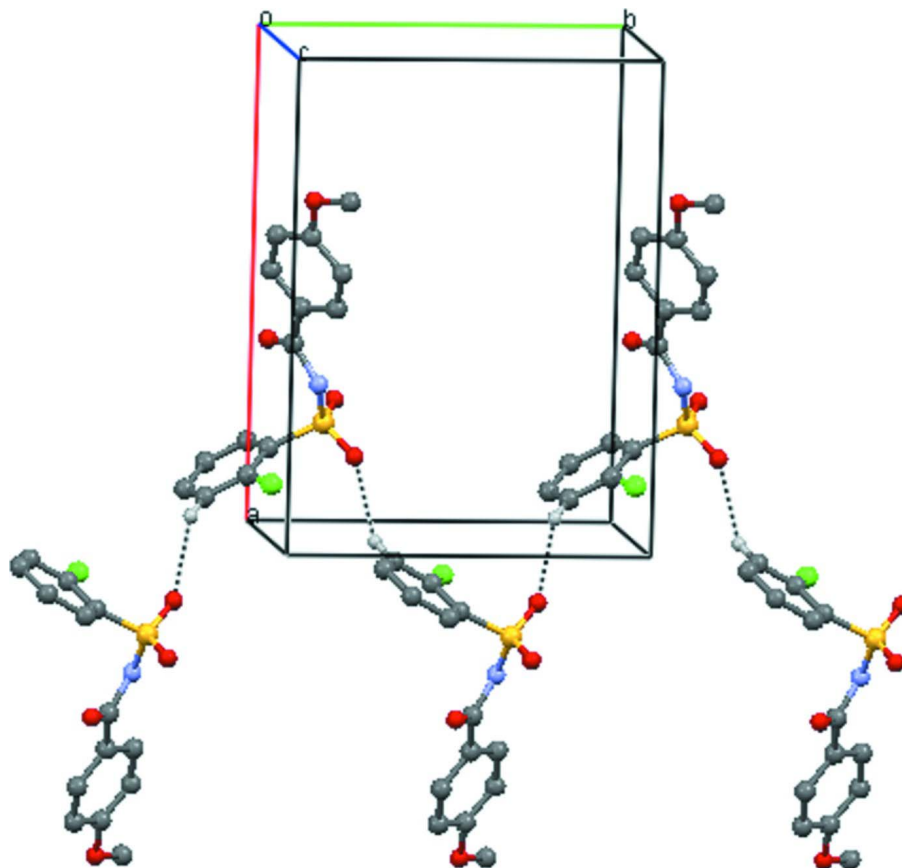


Figure 3

Formation of C(6) chains through C3—H3...O1 interactions running along [001].

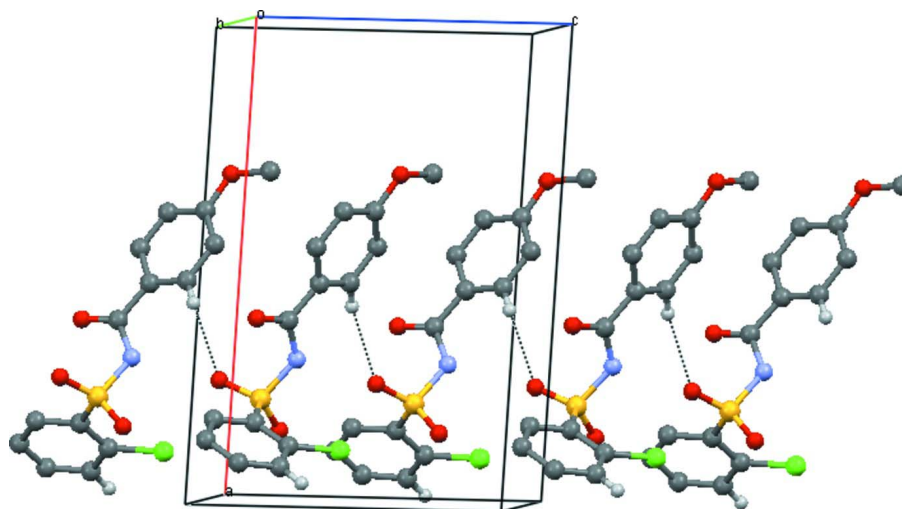


Figure 4

Formation of C(7) chains through C9—H9...O2 interactions running along [001].

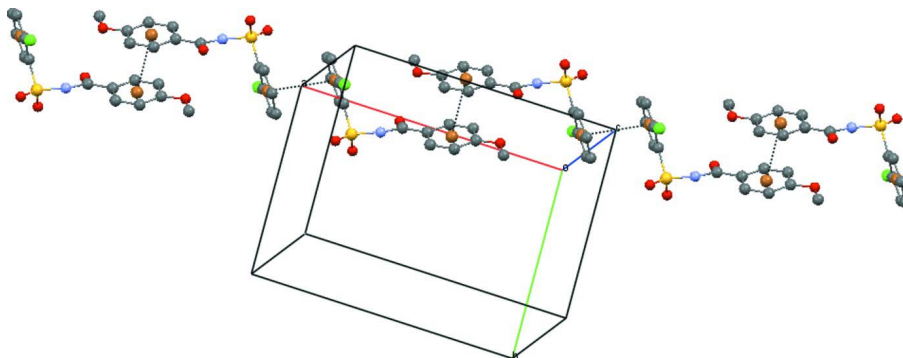


Figure 5

$\pi$ - $\pi$  stacking interactions observed in the crystal structure of I.

## 2-Chloro-*N*-(4-methoxybenzoyl)benzenesulfonamide

### Crystal data

$C_{14}H_{12}ClNO_4S$

$M_r = 325.76$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 14.5293$  (19) Å

$b = 10.6225$  (14) Å

$c = 9.6918$  (14) Å

$\beta = 92.695$  (5)°

$V = 1494.2$  (4) Å<sup>3</sup>

$Z = 4$

$F(000) = 672$

Prism

$D_x = 1.448$  Mg m<sup>-3</sup>

Melting point: 445 K

Cu  $K\alpha$  radiation,  $\lambda = 1.54178$  Å

Cell parameters from 25 reflections

$\theta = 3.0$ – $64.6$ °

$\mu = 3.71$  mm<sup>-1</sup>

$T = 293$  K

Prism, colourless

$0.44 \times 0.35 \times 0.26$  mm

### Data collection

Bruker APEXII CCD  
diffractometer

Radiation source: fine-focus sealed tube  
Graphite monochromator

$\phi$  and  $\psi$  scans

Absorption correction: multi-scan  
(*SADABS*; Bruker, 2009)

$T_{\min} = 0.263$ ,  $T_{\max} = 0.381$

17846 measured reflections

2449 independent reflections

2295 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.058$

$\theta_{\max} = 64.6$ °,  $\theta_{\min} = 3.0$ °

$h = -16 \rightarrow 16$

$k = -12 \rightarrow 12$

$l = -11 \rightarrow 9$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.063$

$wR(F^2) = 0.163$

$S = 1.11$

2449 reflections

195 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

$w = 1/[\sigma^2(F_o^2) + (0.109P)^2 + 0.3886P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.52$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.37$  e Å<sup>-3</sup>

*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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
H1	0.718 (2)	0.192 (3)	0.295 (4)	0.066 (10)*
S1	0.79503 (4)	0.19067 (5)	0.11546 (6)	0.0448 (3)
Cl1	0.90252 (7)	0.02535 (9)	0.35628 (9)	0.0882 (4)
O1	0.85786 (12)	0.27002 (19)	0.1915 (2)	0.0592 (5)
O2	0.75657 (13)	0.2343 (2)	-0.01265 (19)	0.0645 (5)
O4	0.32115 (14)	0.1078 (2)	0.5017 (3)	0.0826 (7)
C9	0.54708 (16)	0.1902 (2)	0.3736 (3)	0.0497 (6)
H9	0.5956	0.2456	0.3921	0.060*
C8	0.55277 (15)	0.1039 (2)	0.2671 (2)	0.0439 (5)
C7	0.63122 (16)	0.0964 (2)	0.1759 (2)	0.0467 (6)
N1	0.71064 (13)	0.1640 (2)	0.2183 (2)	0.0482 (5)
O3	0.62999 (14)	0.0385 (2)	0.0682 (2)	0.0705 (6)
C10	0.47035 (18)	0.1952 (3)	0.4531 (3)	0.0562 (7)
H10	0.4673	0.2543	0.5235	0.067*
C13	0.47841 (19)	0.0229 (3)	0.2399 (3)	0.0603 (7)
H13	0.4801	-0.0340	0.1671	0.072*
C1	0.84769 (15)	0.0440 (2)	0.0838 (3)	0.0485 (6)
C11	0.39843 (17)	0.1122 (3)	0.4276 (3)	0.0565 (6)
C2	0.89668 (17)	-0.0230 (3)	0.1868 (3)	0.0604 (7)
C12	0.4026 (2)	0.0265 (3)	0.3197 (4)	0.0679 (8)
H12	0.3539	-0.0287	0.3013	0.081*
C3	0.9432 (2)	-0.1334 (3)	0.1497 (5)	0.0842 (12)
H3	0.9776	-0.1787	0.2160	0.101*
C6	0.8439 (2)	0.0008 (3)	-0.0515 (3)	0.0638 (7)
H6	0.8105	0.0453	-0.1195	0.077*
C4	0.9373 (3)	-0.1740 (3)	0.0131 (5)	0.0893 (12)
H4	0.9668	-0.2481	-0.0107	0.107*
C5	0.8897 (3)	-0.1082 (4)	-0.0854 (5)	0.0851 (11)
H5	0.8877	-0.1362	-0.1765	0.102*
C14	0.3152 (3)	0.1932 (4)	0.6139 (5)	0.0940 (13)
H14A	0.3676	0.1823	0.6768	0.141*
H14B	0.2598	0.1771	0.6612	0.141*
H14C	0.3141	0.2780	0.5795	0.141*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S1	0.0411 (4)	0.0523 (4)	0.0410 (4)	0.0020 (2)	0.0010 (3)	0.0046 (2)
C11	0.0929 (6)	0.0998 (7)	0.0690 (6)	-0.0016 (5)	-0.0274 (4)	0.0253 (4)
O1	0.0552 (10)	0.0589 (10)	0.0636 (12)	-0.0150 (8)	0.0034 (8)	-0.0062 (8)
O2	0.0623 (11)	0.0852 (13)	0.0459 (10)	0.0168 (10)	0.0013 (8)	0.0179 (9)
O4	0.0488 (11)	0.0906 (16)	0.1105 (19)	-0.0120 (10)	0.0257 (11)	-0.0087 (13)
C9	0.0382 (12)	0.0555 (14)	0.0552 (15)	-0.0069 (9)	-0.0011 (10)	-0.0066 (10)
C8	0.0404 (11)	0.0450 (12)	0.0459 (12)	-0.0027 (9)	-0.0037 (9)	0.0013 (10)
C7	0.0436 (12)	0.0541 (13)	0.0418 (13)	0.0007 (10)	-0.0048 (9)	-0.0023 (10)
N1	0.0421 (10)	0.0628 (12)	0.0394 (12)	-0.0026 (9)	-0.0010 (8)	-0.0064 (10)
O3	0.0594 (11)	0.0961 (15)	0.0557 (12)	-0.0101 (10)	0.0010 (9)	-0.0290 (11)
C10	0.0429 (13)	0.0642 (16)	0.0616 (16)	-0.0030 (10)	0.0047 (11)	-0.0110 (12)
C13	0.0529 (14)	0.0595 (15)	0.0681 (17)	-0.0110 (12)	-0.0015 (12)	-0.0141 (13)
C1	0.0368 (11)	0.0534 (13)	0.0555 (14)	-0.0019 (9)	0.0043 (10)	0.0023 (11)
C11	0.0406 (12)	0.0594 (15)	0.0697 (17)	-0.0029 (11)	0.0055 (11)	0.0053 (13)
C2	0.0396 (12)	0.0604 (16)	0.0806 (19)	-0.0003 (11)	-0.0018 (12)	0.0167 (13)
C12	0.0467 (14)	0.0664 (17)	0.091 (2)	-0.0180 (12)	0.0039 (13)	-0.0085 (15)
C3	0.0488 (15)	0.0647 (19)	0.139 (4)	0.0075 (13)	0.0061 (18)	0.030 (2)
C6	0.0598 (15)	0.0699 (17)	0.0627 (17)	-0.0006 (13)	0.0115 (13)	-0.0087 (14)
C4	0.072 (2)	0.066 (2)	0.133 (4)	0.0070 (16)	0.032 (2)	-0.012 (2)
C5	0.081 (2)	0.077 (2)	0.100 (3)	-0.0035 (18)	0.030 (2)	-0.025 (2)
C14	0.0640 (19)	0.103 (3)	0.119 (3)	0.0030 (18)	0.041 (2)	-0.016 (2)

*Geometric parameters (Å, °)*

S1—O2	1.4154 (19)	C13—C12	1.375 (4)
S1—O1	1.4227 (19)	C13—H13	0.9300
S1—N1	1.641 (2)	C1—C6	1.387 (4)
S1—C1	1.769 (3)	C1—C2	1.394 (4)
C11—C2	1.720 (3)	C11—C12	1.390 (5)
O4—C11	1.362 (3)	C2—C3	1.408 (5)
O4—C14	1.422 (5)	C12—H12	0.9300
C9—C10	1.385 (4)	C3—C4	1.391 (6)
C9—C8	1.386 (4)	C3—H3	0.9300
C9—H9	0.9300	C6—C5	1.383 (5)
C8—C13	1.396 (3)	C6—H6	0.9300
C8—C7	1.477 (3)	C4—C5	1.348 (6)
C7—O3	1.211 (3)	C4—H4	0.9300
C7—N1	1.404 (3)	C5—H5	0.9300
N1—H1	0.80 (4)	C14—H14A	0.9600
C10—C11	1.381 (4)	C14—H14B	0.9600
C10—H10	0.9300	C14—H14C	0.9600
O2—S1—O1	118.55 (13)	O4—C11—C10	124.3 (3)
O2—S1—N1	108.28 (11)	O4—C11—C12	116.2 (2)
O1—S1—N1	105.51 (11)	C10—C11—C12	119.6 (2)

O2—S1—C1	106.98 (13)	C1—C2—C3	118.5 (3)
O1—S1—C1	109.85 (12)	C1—C2—C11	122.2 (2)
N1—S1—C1	107.16 (11)	C3—C2—C11	119.3 (3)
C11—O4—C14	117.6 (2)	C13—C12—C11	120.4 (2)
C10—C9—C8	121.2 (2)	C13—C12—H12	119.8
C10—C9—H9	119.4	C11—C12—H12	119.8
C8—C9—H9	119.4	C4—C3—C2	119.5 (3)
C9—C8—C13	118.4 (2)	C4—C3—H3	120.2
C9—C8—C7	123.8 (2)	C2—C3—H3	120.2
C13—C8—C7	117.8 (2)	C5—C6—C1	120.3 (3)
O3—C7—N1	119.4 (2)	C5—C6—H6	119.9
O3—C7—C8	124.2 (2)	C1—C6—H6	119.9
N1—C7—C8	116.4 (2)	C5—C4—C3	121.3 (3)
C7—N1—S1	122.55 (18)	C5—C4—H4	119.4
C7—N1—H1	122 (2)	C3—C4—H4	119.4
S1—N1—H1	115 (2)	C4—C5—C6	120.1 (4)
C11—C10—C9	119.9 (2)	C4—C5—H5	119.9
C11—C10—H10	120.1	C6—C5—H5	119.9
C9—C10—H10	120.1	O4—C14—H14A	109.5
C12—C13—C8	120.6 (3)	O4—C14—H14B	109.5
C12—C13—H13	119.7	H14A—C14—H14B	109.5
C8—C13—H13	119.7	O4—C14—H14C	109.5
C6—C1—C2	120.3 (3)	H14A—C14—H14C	109.5
C6—C1—S1	117.2 (2)	H14B—C14—H14C	109.5
C2—C1—S1	122.4 (2)		
C10—C9—C8—C13	1.0 (4)	N1—S1—C1—C2	-67.4 (2)
C10—C9—C8—C7	178.0 (2)	C14—O4—C11—C10	-1.0 (5)
C9—C8—C7—O3	-165.9 (3)	C14—O4—C11—C12	179.3 (3)
C13—C8—C7—O3	11.1 (4)	C9—C10—C11—O4	178.5 (3)
C9—C8—C7—N1	12.8 (3)	C9—C10—C11—C12	-1.8 (4)
C13—C8—C7—N1	-170.2 (2)	C6—C1—C2—C3	1.1 (4)
O3—C7—N1—S1	12.0 (3)	S1—C1—C2—C3	-174.5 (2)
C8—C7—N1—S1	-166.74 (17)	C6—C1—C2—C11	-179.3 (2)
O2—S1—N1—C7	47.2 (2)	S1—C1—C2—C11	5.1 (3)
O1—S1—N1—C7	175.11 (19)	C8—C13—C12—C11	0.9 (5)
C1—S1—N1—C7	-67.9 (2)	O4—C11—C12—C13	-179.3 (3)
C8—C9—C10—C11	0.8 (4)	C10—C11—C12—C13	0.9 (5)
C9—C8—C13—C12	-1.9 (4)	C1—C2—C3—C4	-1.5 (4)
C7—C8—C13—C12	-179.0 (3)	C11—C2—C3—C4	178.9 (3)
O2—S1—C1—C6	0.9 (2)	C2—C1—C6—C5	-0.8 (4)
O1—S1—C1—C6	-129.0 (2)	S1—C1—C6—C5	175.0 (2)
N1—S1—C1—C6	116.9 (2)	C2—C3—C4—C5	1.6 (5)
O2—S1—C1—C2	176.7 (2)	C3—C4—C5—C6	-1.3 (6)
O1—S1—C1—C2	46.8 (2)	C1—C6—C5—C4	0.9 (5)



*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N1—H1 $\cdots$ O2 <sup>i</sup>	0.81 (3)	2.09 (3)	2.872 (3)	172 (3)
C3—H3 $\cdots$ O1 <sup>ii</sup>	0.93	2.57	3.370 (4)	144
C9—H9 $\cdots$ O2 <sup>i</sup>	0.93	2.48	3.288 (3)	145

Symmetry codes: (i)  $x, -y+1/2, z+1/2$ ; (ii)  $-x+2, y-1/2, -z+1/2$ .