

2,5-Dimethoxy-*N*-phenylbenzenesulfonamide

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Received 22 February 2017

Accepted 24 February 2017

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

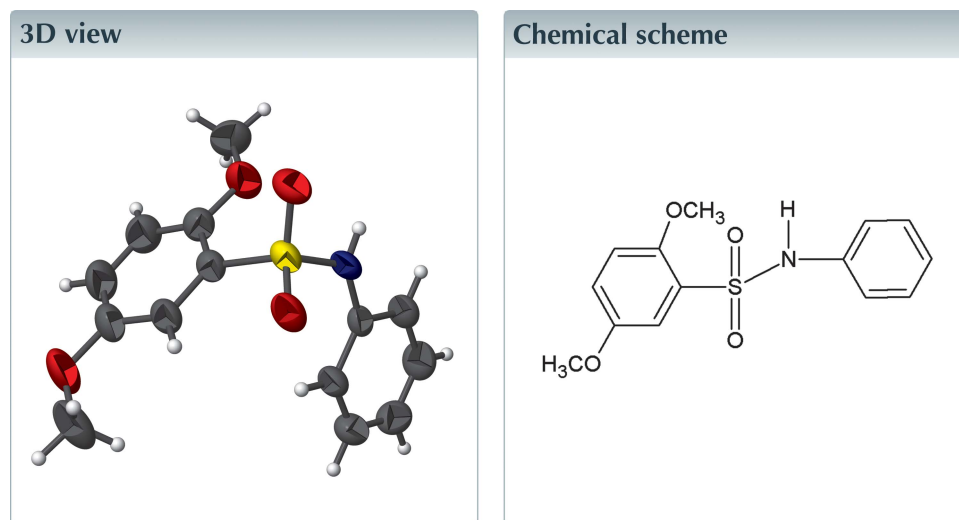
‡ These authors contributed equally

Keywords: crystal structure; sulfonamides; N—H···O hydrogen bonds; intramolecular C—H···O hydrogen bond.

CCDC reference: 1534581

Structural data: full structural data are available from iucrdata.iucr.org

The molecule of the title compound, C₁₄H₁₅NO₄S, is L-shaped, with the central C—S—N—C segment having a torsion angle of $-62.9(2)^\circ$. The dihedral angle between the benzene rings is $89.17(9)^\circ$. The C atoms of the methoxy groups are close to coplanar with their attached benzene ring [deviations = $0.084(4)$ and $-0.192(5)$ Å]. An intramolecular C—H···O hydrogen bond occurs. In the crystal, inversion dimers linked by pairs of N—H···O(S) hydrogen bonds generate $R_2^2(8)$ loops.



Structure description

In recent years, extensive research has been carried out on the synthesis and evaluation of the pharmacological activities of molecules containing the sulfonamide moiety (Mohan *et al.*, 2013). As part of our ongoing studies on sulfonamides (Suchetan *et al.*, 2016), we report herein the crystal structure of the title compound.

The molecule of the title compound (Fig. 1) is L-shaped with the central C1—S1—N1—C7 segment having a torsion angle of $-62.9(2)^\circ$ and the dihedral angle between the benzene rings being $89.17(9)^\circ$. The two methoxy groups are close to coplanar with their attached benzene ring, the values of the torsion angles of the segment C1—C2—O3—C13 and C6—C5—O4—C14, respectively, being $-175.2(3)$ and $-6.0(5)^\circ$. The molecular conformation is consolidated by an intramolecular C12—H12···O1 hydrogen bond (Table 1) forming a closed $S(6)$ motif.

The crystal structure features inversion-related $R_2^2(8)$ dimers linked by pairs of N1—H1···O2ⁱ (Table 1 and Fig. 2) hydrogen bonds.

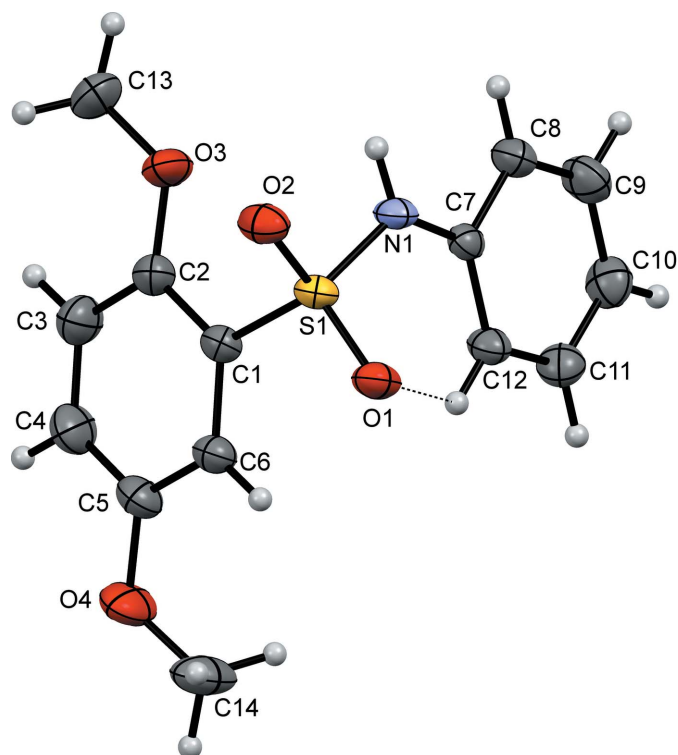


Figure 1
A view of the molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level. The intramolecular hydrogen bond is indicated by a dashed line.

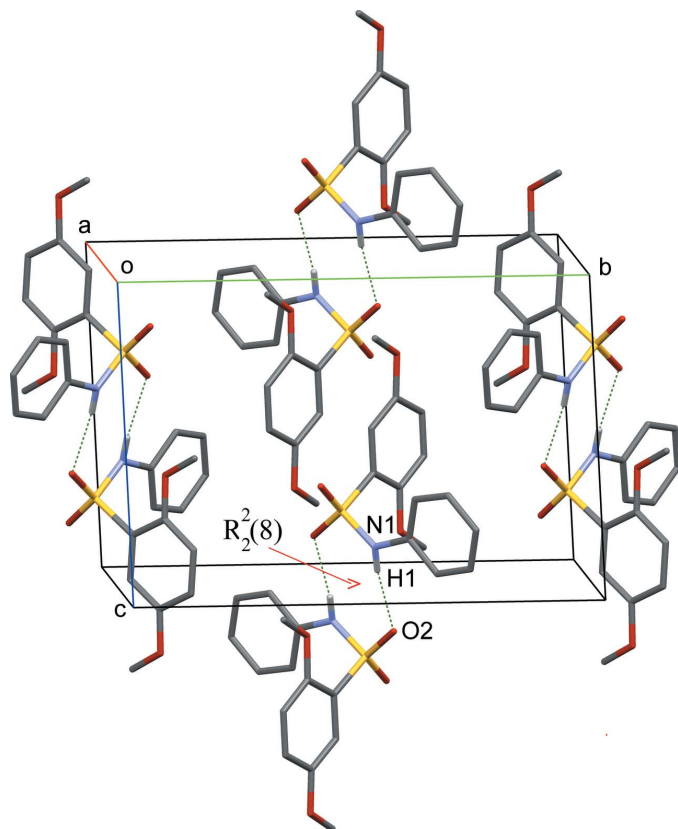


Figure 2
A view of the $R_2^2(8)$ loops displayed in the crystal structure of the title compound due to N–H...O hydrogen bonds (dashed lines, see Table 1).

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C12–H12...O1	0.93	2.52	3.1128	121
N1–H1...O2 ⁱ	0.85	2.17	3.0116 (10)	170

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{15}NO_4S$
M_r	293.33
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	10.4328 (12), 14.3157 (15), 10.4869 (12)
β (°)	117.064 (6)
V (Å ³)	1394.7 (3)
Z	4
Radiation type	Cu $K\alpha$
μ (mm ⁻¹)	2.19
Crystal size (mm)	0.28 × 0.22 × 0.17
Data collection	
Diffractometer	Bruker APEXII
Absorption correction	Multi-scan (SADABS; Bruker, 2009)
T_{min}, T_{max}	0.589, 0.689
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10083, 2305, 1943
R_{int}	0.065
$(\sin \theta/\lambda)_{max}$ (Å ⁻¹)	0.586
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.067, 0.186, 1.04
No. of reflections	2305
No. of parameters	188
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å ⁻³)	0.42, -0.33

Computer programs: APEX2, SAINT-Plus and XPREP (Bruker, 2009), SHELXT2016 (Sheldrick, 2015a), SHELXL2016 (Sheldrick, 2015b) and Mercury (Macrae *et al.*, 2008).

Synthesis and crystallization

The title compound was prepared according to a literature method (Vinola *et al.*, 2015). The purity of the compound was checked by determining its melting point. Prismatic single crystals suitable for X-ray diffraction study were obtained by slow evaporation of an ethanolic solution of the compound at room temperature.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors are thankful to the Institution of Excellence, Vijnana Bhavana, University of Mysore, Mysore, for providing

the single-crystal X-ray diffraction data. KS is thankful to the University Grants Commission (UGC), New Delhi for the financial assistance under its MRP scheme.

References

- Bruker (2009). *APEX2, SADABS, SAINT-Plus and XPREP*. Bruker AXS Inc., Madison, Wisconsin, USA.
- 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.
- Mohan, N. R., Sreenivasa, S., Manojkumar, K. E. & Chakrapani Rao, T. M. (2013). *J. Appl. Chem.* **2**, 722–729.
- Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
- Suchetan, P. A., Naveen, S., Lokanath, N. K., Srivishnu, K. S., Supriya, G. M. & Lakshmikantha, H. N. (2016). *Acta Cryst.* **E72**, 575–582.
- Vinola, Z. R., Snehal, Naveen, S., Lokanath, N. K. & Suchetan, P. A. (2015). *Pharma Chem.* **7**, 299–307.

full crystallographic data

IUCrData (2017). **2**, x170311 [<https://doi.org/10.1107/S241431461700311X>]

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2,5-Dimethoxy-*N*-phenylbenzenesulfonamide*Crystal data*

$C_{14}H_{15}NO_4S$

$M_r = 293.33$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 10.4328$ (12) Å

$b = 14.3157$ (15) Å

$c = 10.4869$ (12) Å

$\beta = 117.064$ (6)°

$V = 1394.7$ (3) Å³

$Z = 4$

$F(000) = 616$

Prism

$D_x = 1.397$ Mg m⁻³

Melting point: 439 K

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

Cell parameters from 143 reflections

$\theta = 7.8$ – 64.5 °

$\mu = 2.19$ mm⁻¹

$T = 296$ K

Prism, colourless

$0.28 \times 0.22 \times 0.17$ mm

Data collection

Bruker APEXII

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

phi and φ scans

Absorption correction: multi-scan

(SADABS; Bruker, 2009)

$T_{\min} = 0.589$, $T_{\max} = 0.689$

10083 measured reflections

2305 independent reflections

1943 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.065$

$\theta_{\max} = 64.5$ °, $\theta_{\min} = 7.8$ °

$h = -12 \rightarrow 11$

$k = -16 \rightarrow 16$

$l = -12 \rightarrow 11$

1 standard reflections every 1 reflections

intensity decay: 0.1%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.067$

$wR(F^2) = 0.186$

$S = 1.04$

2305 reflections

188 parameters

1 restraint

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.1364P)^2]$

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

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

$\Delta\rho_{\max} = 0.42$ e Å⁻³

$\Delta\rho_{\min} = -0.33$ e Å⁻³

Extinction correction: SHELXL2016

(Sheldrick, 2015b),

$F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0106 (19)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3763 (3)	0.95415 (17)	0.1449 (3)	0.0446 (6)
C2	0.4926 (3)	0.8928 (2)	0.2056 (3)	0.0554 (7)
C3	0.5215 (4)	0.8363 (2)	0.1138 (4)	0.0729 (9)
H3	0.598056	0.794521	0.151637	0.087*
C4	0.4387 (4)	0.8413 (3)	−0.0311 (4)	0.0770 (10)
H4	0.460099	0.803009	−0.090500	0.092*
C5	0.3243 (3)	0.9021 (2)	−0.0908 (3)	0.0629 (8)
C6	0.2922 (3)	0.95933 (18)	−0.0021 (3)	0.0517 (7)
H6	0.215095	1.000645	−0.040936	0.062*
C7	0.1886 (3)	0.89741 (16)	0.3259 (3)	0.0408 (6)
C8	0.1959 (3)	0.8411 (2)	0.4360 (3)	0.0560 (7)
H8	0.272972	0.846112	0.527111	0.067*
C9	0.0868 (4)	0.7771 (2)	0.4089 (4)	0.0691 (9)
H9	0.090240	0.739609	0.482882	0.083*
C10	−0.0254 (3)	0.7683 (2)	0.2754 (3)	0.0642 (8)
H10	−0.097532	0.724621	0.258041	0.077*
C11	−0.0312 (3)	0.8241 (2)	0.1673 (3)	0.0592 (7)
H11	−0.108127	0.818391	0.076232	0.071*
C12	0.0749 (3)	0.88843 (18)	0.1910 (3)	0.0490 (7)
H12	0.070090	0.925756	0.116314	0.059*
C13	0.6919 (3)	0.8361 (3)	0.4161 (4)	0.0818 (10)
H13A	0.666377	0.771490	0.394795	0.123*
H13B	0.736012	0.845323	0.517872	0.123*
H13C	0.758229	0.853612	0.380173	0.123*
C14	0.1182 (5)	0.9532 (3)	−0.3002 (4)	0.0897 (12)
H14A	0.056384	0.935813	−0.258691	0.135*
H14B	0.070058	0.941372	−0.401306	0.135*
H14C	0.140914	1.018497	−0.283666	0.135*
N1	0.3015 (3)	0.96370 (15)	0.3628 (2)	0.0477 (6)
O1	0.2068 (2)	1.07776 (12)	0.16321 (19)	0.0545 (6)
O2	0.4588 (2)	1.08222 (13)	0.3458 (2)	0.0583 (6)
O3	0.5672 (2)	0.89154 (16)	0.3507 (2)	0.0716 (7)
O4	0.2466 (3)	0.90022 (19)	−0.2364 (2)	0.0871 (8)
S1	0.33445 (6)	1.02855 (4)	0.25483 (6)	0.0437 (3)
H1	0.375 (2)	0.9564 (18)	0.443 (2)	0.050 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0469 (15)	0.0512 (13)	0.0360 (14)	0.0014 (11)	0.0191 (12)	0.0032 (10)
C2	0.0495 (15)	0.0704 (17)	0.0467 (16)	0.0083 (13)	0.0223 (13)	0.0076 (13)
C3	0.0640 (19)	0.087 (2)	0.068 (2)	0.0248 (17)	0.0306 (17)	0.0030 (16)
C4	0.077 (2)	0.100 (2)	0.062 (2)	0.0176 (19)	0.0388 (18)	-0.0133 (17)
C5	0.070 (2)	0.0805 (19)	0.0414 (17)	0.0047 (16)	0.0285 (15)	-0.0040 (13)
C6	0.0559 (17)	0.0604 (15)	0.0372 (15)	0.0038 (12)	0.0199 (13)	0.0033 (11)
C7	0.0440 (13)	0.0473 (13)	0.0334 (13)	0.0067 (10)	0.0196 (10)	-0.0005 (10)
C8	0.0577 (16)	0.0681 (17)	0.0401 (15)	0.0002 (13)	0.0204 (13)	0.0067 (12)
C9	0.080 (2)	0.0732 (19)	0.0603 (19)	-0.0054 (16)	0.0372 (17)	0.0132 (15)
C10	0.0610 (18)	0.0627 (17)	0.071 (2)	-0.0115 (14)	0.0316 (16)	-0.0060 (14)
C11	0.0561 (16)	0.0654 (17)	0.0498 (17)	-0.0063 (13)	0.0186 (13)	-0.0098 (13)
C12	0.0509 (15)	0.0578 (15)	0.0345 (14)	-0.0051 (12)	0.0161 (11)	-0.0023 (11)
C13	0.0550 (19)	0.107 (3)	0.072 (2)	0.0266 (18)	0.0196 (16)	0.0222 (19)
C14	0.110 (3)	0.102 (3)	0.0386 (18)	0.032 (2)	0.018 (2)	0.0003 (18)
N1	0.0444 (13)	0.0642 (14)	0.0255 (12)	-0.0043 (10)	0.0079 (10)	0.0028 (9)
O1	0.0630 (12)	0.0582 (11)	0.0360 (10)	0.0137 (9)	0.0170 (9)	0.0046 (7)
O2	0.0625 (12)	0.0601 (11)	0.0430 (11)	-0.0144 (9)	0.0160 (9)	-0.0012 (8)
O3	0.0617 (13)	0.0988 (16)	0.0441 (12)	0.0310 (11)	0.0151 (10)	0.0137 (10)
O4	0.0956 (18)	0.122 (2)	0.0404 (12)	0.0229 (15)	0.0278 (12)	-0.0134 (12)
S1	0.0481 (5)	0.0484 (5)	0.0295 (5)	0.0005 (2)	0.0130 (3)	0.0020 (2)

Geometric parameters (Å, °)

C1—C6	1.386 (4)	C9—H9	0.9300
C1—C2	1.395 (4)	C10—C11	1.366 (4)
C1—S1	1.764 (3)	C10—H10	0.9300
C2—O3	1.359 (3)	C11—C12	1.373 (4)
C2—C3	1.391 (4)	C11—H11	0.9300
C3—C4	1.366 (5)	C12—H12	0.9300
C3—H3	0.9300	C13—O3	1.407 (4)
C4—C5	1.376 (4)	C13—H13A	0.9600
C4—H4	0.9300	C13—H13B	0.9600
C5—O4	1.365 (4)	C13—H13C	0.9600
C5—C6	1.390 (4)	C14—O4	1.416 (4)
C6—H6	0.9300	C14—H14A	0.9600
C7—C12	1.378 (4)	C14—H14B	0.9600
C7—C8	1.382 (4)	C14—H14C	0.9600
C7—N1	1.422 (3)	N1—S1	1.618 (2)
C8—C9	1.387 (4)	N1—H1	0.849 (18)
C8—H8	0.9300	O1—S1	1.4240 (19)
C9—C10	1.362 (4)	O2—S1	1.4345 (19)
C6—C1—C2	121.3 (2)	C10—C11—C12	121.0 (3)
C6—C1—S1	118.3 (2)	C10—C11—H11	119.5
C2—C1—S1	120.3 (2)	C12—C11—H11	119.5

O3—C2—C3	125.0 (3)	C11—C12—C7	119.7 (2)
O3—C2—C1	117.1 (2)	C11—C12—H12	120.2
C3—C2—C1	117.9 (3)	C7—C12—H12	120.2
C4—C3—C2	120.8 (3)	O3—C13—H13A	109.5
C4—C3—H3	119.6	O3—C13—H13B	109.5
C2—C3—H3	119.6	H13A—C13—H13B	109.5
C3—C4—C5	121.2 (3)	O3—C13—H13C	109.5
C3—C4—H4	119.4	H13A—C13—H13C	109.5
C5—C4—H4	119.4	H13B—C13—H13C	109.5
O4—C5—C4	116.5 (3)	O4—C14—H14A	109.5
O4—C5—C6	124.0 (3)	O4—C14—H14B	109.5
C4—C5—C6	119.4 (3)	H14A—C14—H14B	109.5
C1—C6—C5	119.3 (3)	O4—C14—H14C	109.5
C1—C6—H6	120.3	H14A—C14—H14C	109.5
C5—C6—H6	120.3	H14B—C14—H14C	109.5
C12—C7—C8	119.9 (2)	C7—N1—S1	127.30 (18)
C12—C7—N1	124.1 (2)	C7—N1—H1	116.7 (19)
C8—C7—N1	116.0 (2)	S1—N1—H1	113 (2)
C7—C8—C9	119.1 (3)	C2—O3—C13	119.0 (2)
C7—C8—H8	120.4	C5—O4—C14	118.0 (2)
C9—C8—H8	120.4	O1—S1—O2	117.96 (12)
C10—C9—C8	120.9 (3)	O1—S1—N1	108.95 (12)
C10—C9—H9	119.6	O2—S1—N1	104.93 (11)
C8—C9—H9	119.6	O1—S1—C1	106.93 (11)
C9—C10—C11	119.5 (3)	O2—S1—C1	109.90 (12)
C9—C10—H10	120.3	N1—S1—C1	107.79 (13)
C11—C10—H10	120.3		
C6—C1—C2—O3	-178.9 (3)	C10—C11—C12—C7	-0.4 (4)
S1—C1—C2—O3	2.0 (3)	C8—C7—C12—C11	0.6 (4)
C6—C1—C2—C3	-0.4 (4)	N1—C7—C12—C11	-177.9 (2)
S1—C1—C2—C3	-179.5 (2)	C12—C7—N1—S1	-10.5 (4)
O3—C2—C3—C4	178.8 (3)	C8—C7—N1—S1	170.9 (2)
C1—C2—C3—C4	0.4 (5)	C3—C2—O3—C13	6.4 (5)
C2—C3—C4—C5	-0.2 (6)	C1—C2—O3—C13	-175.2 (3)
C3—C4—C5—O4	-178.6 (3)	C4—C5—O4—C14	172.4 (3)
C3—C4—C5—C6	-0.1 (5)	C6—C5—O4—C14	-6.0 (5)
C2—C1—C6—C5	0.1 (4)	C7—N1—S1—O1	52.9 (2)
S1—C1—C6—C5	179.2 (2)	C7—N1—S1—O2	-179.9 (2)
O4—C5—C6—C1	178.5 (3)	C7—N1—S1—C1	-62.9 (2)
C4—C5—C6—C1	0.1 (4)	C6—C1—S1—O1	4.3 (2)
C12—C7—C8—C9	-0.9 (4)	C2—C1—S1—O1	-176.6 (2)
N1—C7—C8—C9	177.8 (2)	C6—C1—S1—O2	-124.9 (2)
C7—C8—C9—C10	1.0 (5)	C2—C1—S1—O2	54.3 (2)
C8—C9—C10—C11	-0.7 (5)	C6—C1—S1—N1	121.3 (2)
C9—C10—C11—C12	0.4 (5)	C2—C1—S1—N1	-59.5 (2)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C12—H12···O1	0.93	2.52	3.1128	121
N1—H1···O2 ⁱ	0.85	2.17	3.0116 (10)	170

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