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1-(Phenylsulfonyl)-1*H*-indole-2-carbaldehyde

Leslie W. Pineda,[a,b] Natasha Ferllini[a] and Jorge A. Cabezas[a]*

[a]Escuela de Química, Universidad de Costa Rica, 11501-2060, San José, Costa Rica, and [b]Centro de Electroquímica y Energía Química (CELEQ), Universidad de Costa Rica, 11501-2060, San José, Costa Rica. *Correspondence e-mail: jorge.cabezas@ucr.ac.cr

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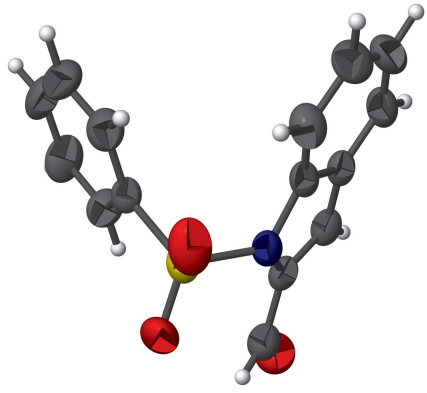
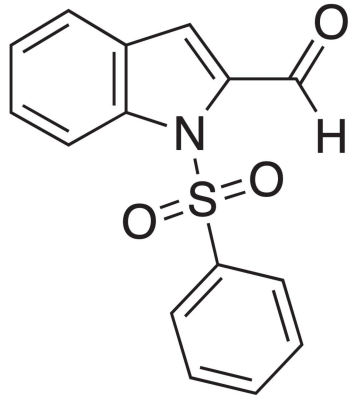
The title compound, $C_{15}H_{11}NO_3S$, was prepared by a facile synthetic approach. The N atom in the pyrrole ring of the indole moiety is pyramidal (bond-angle sum = 350.0°) and the phenyl ring of the phenylsulfonyl motif forms a dihedral angle of 76.24 (7)° with the mean plane of the indole ring system. In the crystal, C—H···O and C—H···π interactions link the molecules into a three-dimensional network.

3D view



Chemical scheme



Structure description

The indole ring framework is a heterocyclic system found in many natural products. Many of these compounds possess biological activity, from neurotransmitter serotonin to vinblastine, an alkaloid clinically used as an anticancer agent (Inman & Moody, 2013). The title compound, **1**, is a useful synthetic intermediate, which has been used in the preparation of bouchardatine, a natural occurring alkaloid isolated from the rutaecarpine family (Naik *et al.*, 2013). It has also been used to synthesize bis(1*H*-indol-2-yl)methanones, potent inhibitors of FLT3 receptor tyrosine kinase (Mahboobi *et al.*, 2006). Usually, this synthetic intermediate is synthesized from indole, which is treated with benzenesulfonyl chloride under basic conditions, and further formylated at the 2-position by sequential treatment with lithium diisopropyl amide and dimethylformamide. As a part of our program of the synthesis of biologically active sulfanilamide derivatives (Cabezas & Arias, 2019), we report herein a straightforward approach for the synthesis of **1** and its crystal structure.

The crystal structure of **1** has monoclinic symmetry with one molecule in the asymmetric unit: the five-membered pyrrole ring of the indole motif contains a carbaldehyde group and also binds *via* a nitrogen atom to a phenylsulfonyl fragment (Fig. 1). The bond lengths and angles in **1** do not show any unexpected features (Palani *et al.*, 2006; Sakthivel *et al.*, 2006). The bond angles O3—S1—O2 [120.63 (10)°] and N1—S1—C15 [104.80 (8)°]

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

*Cg*2 is the centroid of the C3–C8 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···O3	0.93	2.44	3.014 (3)	120
C9—H9···O2	0.93	2.34	2.869 (3)	116
C4—H4···O1^{i}	0.93	2.51	3.343 (3)	150
C12—H12···*Cg*2^{ii}	0.93	2.71	3.638 (3)	174

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

support the distorted tetrahedral geometry around atom S1. Atom N1 within the pyrrole ring deviates from planar geometry, showing a slight pyramidalization (bond-angle sum = 350.0°). The phenyl ring of the phenylsulfonyl motif subtends a dihedral angle of 76.24 (7)° with the mean plane of the indole ring system. There are two short intramolecular C—H···O contacts and the crystal packing features C—H···O and C—H···π interactions (Table 1, Fig. 2).

Synthesis and crystallization

The title compound, **1**, was synthesized by the reaction of 2-iodoaniline, **2**, with benzenesulfonyl chloride, **3**, in the presence of pyridine to obtain after purification by column chromatography, the iodosulfonamide **4**. Treatment of the latter iodide, **4**, with propargyl alcohol, **5**, under Sonogashira's reaction conditions (Sonogashira *et al.*, 1975), at room temperature, produced [1-(phenylsulfonyl)-1*H*-indol-2-yl]methanol **6** in a one-pot reaction and with overall yield of 84%. Similar synthetic strategies, using *N*-(2-iodophenyl)methane sulfonamides, required heating at 100–110°C in a sealed tube (Sakamoto *et al.*, 1988). Oxidation of this alcohol, with pyridinium chlorochromate, provided the target aldehyde in 81% yield (Fig. 3). The product was recrystallized from ethyl acetate solution at room temperature resulting in light-yellow blocks.

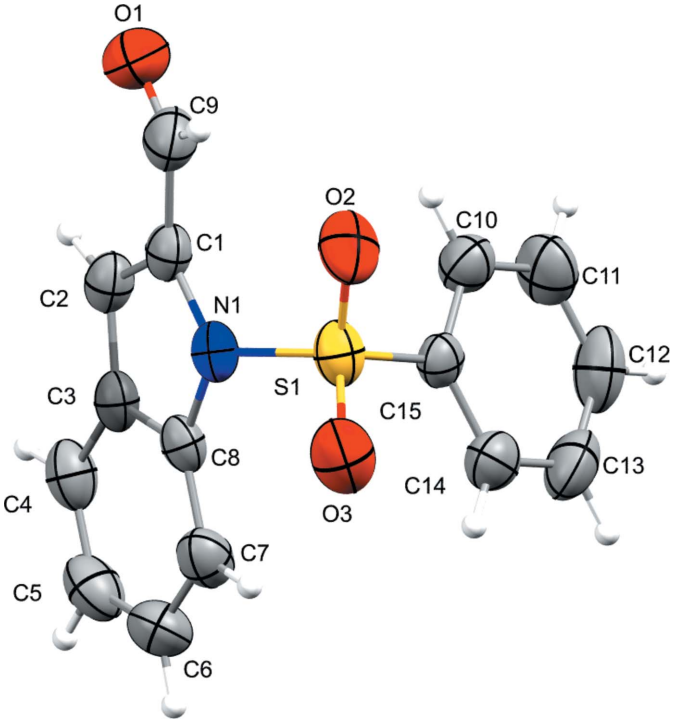

Figure 1
Molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

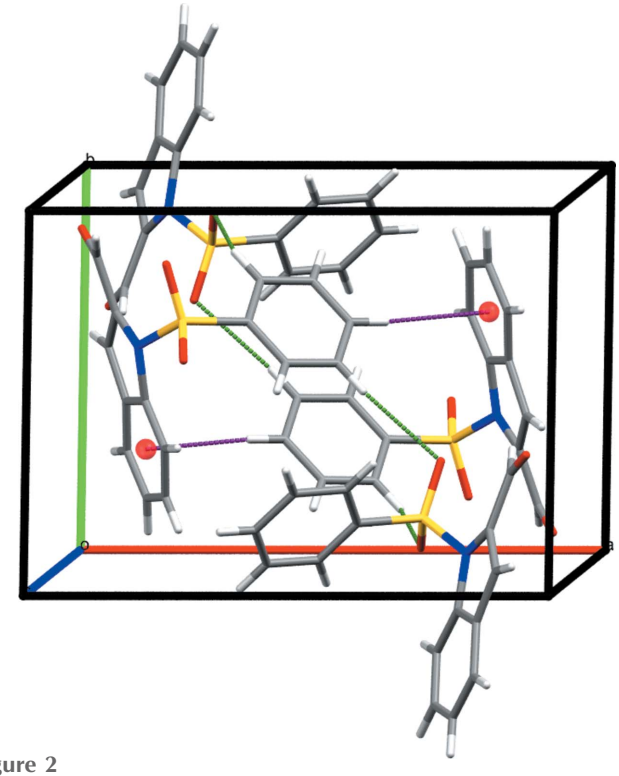

Figure 2
Packing view of the title compound. C—H···O and C—H···π interactions are shown as green and purple dashed lines, respectively.

Refinement

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

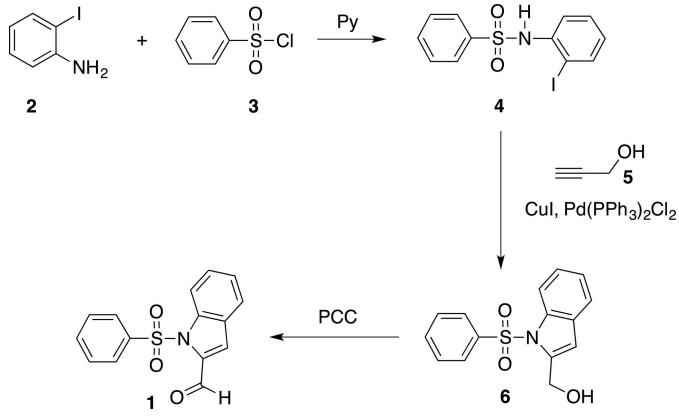

Figure 3
A synthetic scheme for the preparation of the title compound.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{11}NO_3S$
M_r	285.31
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	273
a, b, c (Å)	12.6886 (7), 9.2655 (6), 11.6024 (7)
β (°)	105.374 (2)
V (Å^3)	1315.24 (14)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.25
Crystal size (mm)	0.20 × 0.15 × 0.15
Data collection	
Diffractometer	Bruker D8 Venture
Absorption correction	Multi-scan (*SADABS*; Bruker, 2015)
T_{min}, T_{max}	0.690, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	18696, 3032, 1791
R_{int}	0.057
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.651
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.048, 0.114, 1.01
No. of reflections	3032
No. of parameters	181
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.24, −0.33

Computer programs: *APEX3* and *SAINT* (Bruker, 2015), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2020) and *publCIF* (Westrip, 2010).

Funding information

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full crystallographic data

IUCrData (2022). **7**, x220401 [https://doi.org/10.1107/S2414314622004011]

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Crystal data

$C_{15}H_{11}NO_3S$
M_r = 285.31
Monoclinic, $P2_1/c$
a = 12.6886 (7) Å
b = 9.2655 (6) Å
c = 11.6024 (7) Å
β = 105.374 (2)°
V = 1315.24 (14) Å^3
Z = 4
$F(000)$ = 592
D_x = 1.441 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3972 reflections
θ = 2.8–23.8°
μ = 0.25 mm^{-1}
T = 273 K
Block, clear light yellow
0.20 × 0.15 × 0.15 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: Incoatec Microsource
Mirrors monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2015)
T_{min} = 0.690, T_{max} = 0.746
18696 measured reflections
3032 independent reflections
1791 reflections with $I > 2\sigma(I)$
R_{int} = 0.057
θ_{max} = 27.5°, θ_{min} = 2.8°
h = −16→16
k = −12→12
l = −15→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.114
S = 1.01
3032 reflections
181 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0529P)^2 + 0.1517P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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.

Refinement. All hydrogen atoms were placed geometrically and refined using a riding-model approximation, with C—H = 0.95–1.00 Å and U_{iso}(H) = 1.2U_{eq}(C).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.26292 (4)	0.67883 (6)	0.70277 (4)	0.0461 (2)
O1	0.03455 (14)	0.88036 (19)	0.38393 (17)	0.0771 (5)
O2	0.23104 (12)	0.82550 (16)	0.70663 (14)	0.0621 (5)
O3	0.27691 (13)	0.58878 (18)	0.80466 (12)	0.0645 (5)
N1	0.16726 (12)	0.60075 (18)	0.59334 (13)	0.0407 (4)
C1	0.11260 (15)	0.6739 (2)	0.48512 (18)	0.0421 (5)
C2	0.08938 (16)	0.5776 (2)	0.39588 (18)	0.0447 (5)
H2	0.0523	0.5978	0.317	0.054*
C3	0.13005 (15)	0.4395 (2)	0.44002 (17)	0.0400 (5)
C4	0.12749 (18)	0.3046 (3)	0.3870 (2)	0.0537 (6)
H4	0.0946	0.2926	0.3058	0.064*
C5	0.17400 (19)	0.1897 (3)	0.4559 (2)	0.0599 (7)
H5	0.1735	0.0992	0.4211	0.072*
C6	0.2220 (2)	0.2070 (2)	0.5775 (2)	0.0595 (6)
H6	0.2527	0.1272	0.6226	0.071*
C7	0.22548 (18)	0.3386 (2)	0.63281 (19)	0.0519 (6)
H7	0.2578	0.3491	0.7142	0.062*
C8	0.17904 (15)	0.4546 (2)	0.56285 (17)	0.0384 (5)
C9	0.07306 (18)	0.8234 (3)	0.4781 (2)	0.0586 (6)
H9	0.0781	0.8747	0.5483	0.07*
C10	0.39626 (19)	0.7687 (2)	0.5702 (2)	0.0523 (6)
H10	0.3429	0.8375	0.5392	0.063*
C11	0.4910 (2)	0.7626 (3)	0.5327 (2)	0.0670 (7)
H11	0.5014	0.8276	0.4756	0.08*
C12	0.5693 (2)	0.6617 (3)	0.5788 (2)	0.0704 (8)
H12	0.633	0.6591	0.5537	0.084*
C13	0.55456 (19)	0.5649 (3)	0.6616 (2)	0.0672 (7)
H13	0.6078	0.4958	0.692	0.081*
C14	0.46101 (18)	0.5694 (2)	0.70013 (19)	0.0538 (6)
H14	0.451	0.5038	0.7569	0.065*
C15	0.38264 (15)	0.6712 (2)	0.65436 (16)	0.0385 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0452 (3)	0.0563 (4)	0.0376 (3)	−0.0103 (3)	0.0123 (2)	−0.0122 (3)
O1	0.0678 (12)	0.0615 (11)	0.0899 (14)	0.0065 (9)	−0.0001 (10)	0.0109 (11)
O2	0.0582 (10)	0.0570 (11)	0.0721 (11)	−0.0043 (8)	0.0187 (8)	−0.0292 (8)
O3	0.0745 (11)	0.0880 (13)	0.0322 (8)	−0.0219 (9)	0.0160 (7)	−0.0020 (8)
N1	0.0362 (9)	0.0476 (11)	0.0390 (10)	−0.0071 (8)	0.0112 (8)	−0.0081 (8)
C1	0.0305 (11)	0.0500 (13)	0.0459 (12)	−0.0037 (10)	0.0106 (9)	−0.0006 (11)
C2	0.0355 (11)	0.0593 (14)	0.0388 (12)	−0.0048 (11)	0.0091 (9)	0.0004 (11)
C3	0.0332 (11)	0.0498 (14)	0.0384 (12)	−0.0092 (10)	0.0119 (9)	−0.0058 (10)
C4	0.0502 (14)	0.0605 (16)	0.0506 (13)	−0.0152 (12)	0.0137 (11)	−0.0164 (13)
C5	0.0657 (16)	0.0443 (14)	0.0737 (18)	−0.0122 (12)	0.0253 (14)	−0.0141 (14)

C6	0.0661 (16)	0.0451 (15)	0.0672 (17)	−0.0050 (12)	0.0175 (13)	0.0078 (13)
C7	0.0575 (15)	0.0532 (15)	0.0433 (13)	−0.0113 (12)	0.0105 (11)	0.0025 (12)
C8	0.0367 (11)	0.0423 (13)	0.0392 (12)	−0.0102 (10)	0.0154 (9)	−0.0040 (10)
C9	0.0426 (14)	0.0569 (16)	0.0715 (17)	−0.0014 (12)	0.0070 (12)	−0.0052 (14)
C10	0.0469 (14)	0.0591 (15)	0.0485 (13)	0.0016 (11)	0.0082 (11)	0.0058 (12)
C11	0.0592 (16)	0.0840 (19)	0.0619 (16)	−0.0138 (15)	0.0235 (13)	0.0115 (14)
C12	0.0386 (14)	0.102 (2)	0.0726 (18)	−0.0104 (15)	0.0182 (13)	−0.0179 (17)
C13	0.0404 (14)	0.0761 (18)	0.0761 (18)	0.0096 (13)	−0.0004 (13)	−0.0081 (15)
C14	0.0515 (14)	0.0565 (15)	0.0476 (13)	−0.0034 (12)	0.0028 (11)	0.0012 (11)
C15	0.0360 (11)	0.0449 (12)	0.0313 (10)	−0.0040 (10)	0.0029 (8)	−0.0027 (10)

Geometric parameters (Å, º)

S1—O3	1.4191 (15)	C6—C7	1.373 (3)
S1—O2	1.4220 (16)	C6—H6	0.93
S1—N1	1.6708 (16)	C7—C8	1.382 (3)
S1—C15	1.755 (2)	C7—H7	0.93
O1—C9	1.195 (3)	C9—H9	0.93
N1—C8	1.417 (2)	C10—C15	1.375 (3)
N1—C1	1.434 (2)	C10—C11	1.384 (3)
C1—C2	1.339 (3)	C10—H10	0.93
C1—C9	1.468 (3)	C11—C12	1.366 (4)
C2—C3	1.423 (3)	C11—H11	0.93
C2—H2	0.93	C12—C13	1.362 (3)
C3—C4	1.390 (3)	C12—H12	0.93
C3—C8	1.403 (3)	C13—C14	1.376 (3)
C4—C5	1.367 (3)	C13—H13	0.93
C4—H4	0.93	C14—C15	1.371 (3)
C5—C6	1.390 (3)	C14—H14	0.93
C5—H5	0.93		
O3—S1—O2	120.63 (10)	C6—C7—C8	117.4 (2)
O3—S1—N1	106.48 (9)	C6—C7—H7	121.3
O2—S1—N1	106.40 (9)	C8—C7—H7	121.3
O3—S1—C15	108.37 (10)	C7—C8—C3	121.63 (19)
O2—S1—C15	109.04 (10)	C7—C8—N1	130.82 (18)
N1—S1—C15	104.80 (8)	C3—C8—N1	107.53 (17)
C8—N1—C1	106.96 (15)	O1—C9—C1	121.2 (2)
C8—N1—S1	119.99 (13)	O1—C9—H9	119.4
C1—N1—S1	123.09 (14)	C1—C9—H9	119.4
C2—C1—N1	108.53 (18)	C15—C10—C11	118.7 (2)
C2—C1—C9	125.7 (2)	C15—C10—H10	120.7
N1—C1—C9	125.00 (19)	C11—C10—H10	120.7
C1—C2—C3	109.67 (18)	C12—C11—C10	120.5 (2)
C1—C2—H2	125.2	C12—C11—H11	119.8
C3—C2—H2	125.2	C10—C11—H11	119.8
C4—C3—C8	119.5 (2)	C13—C12—C11	120.4 (2)
C4—C3—C2	133.3 (2)	C13—C12—H12	119.8

C8—C3—C2	107.27 (18)	C11—C12—H12	119.8
C5—C4—C3	119.0 (2)	C12—C13—C14	120.0 (2)
C5—C4—H4	120.5	C12—C13—H13	120.0
C3—C4—H4	120.5	C14—C13—H13	120.0
C4—C5—C6	120.6 (2)	C15—C14—C13	119.6 (2)
C4—C5—H5	119.7	C15—C14—H14	120.2
C6—C5—H5	119.7	C13—C14—H14	120.2
C7—C6—C5	121.9 (2)	C14—C15—C10	120.8 (2)
C7—C6—H6	119.1	C14—C15—S1	120.28 (17)
C5—C6—H6	119.1	C10—C15—S1	118.89 (16)
O3—S1—N1—C8	−53.53 (16)	C4—C3—C8—N1	−178.29 (17)
O2—S1—N1—C8	176.62 (14)	C2—C3—C8—N1	0.5 (2)
C15—S1—N1—C8	61.18 (16)	C1—N1—C8—C7	−179.9 (2)
O3—S1—N1—C1	165.38 (15)	S1—N1—C8—C7	33.4 (3)
O2—S1—N1—C1	35.52 (17)	C1—N1—C8—C3	−1.55 (19)
C15—S1—N1—C1	−79.91 (16)	S1—N1—C8—C3	−148.17 (13)
C8—N1—C1—C2	2.0 (2)	C2—C1—C9—O1	−15.8 (3)
S1—N1—C1—C2	147.38 (14)	N1—C1—C9—O1	175.04 (19)
C8—N1—C1—C9	172.76 (18)	C15—C10—C11—C12	−0.2 (4)
S1—N1—C1—C9	−41.9 (3)	C10—C11—C12—C13	0.7 (4)
N1—C1—C2—C3	−1.7 (2)	C11—C12—C13—C14	−0.7 (4)
C9—C1—C2—C3	−172.36 (19)	C12—C13—C14—C15	0.4 (4)
C1—C2—C3—C4	179.4 (2)	C13—C14—C15—C10	0.1 (3)
C1—C2—C3—C8	0.7 (2)	C13—C14—C15—S1	−179.84 (17)
C8—C3—C4—C5	−0.7 (3)	C11—C10—C15—C14	−0.2 (3)
C2—C3—C4—C5	−179.1 (2)	C11—C10—C15—S1	179.77 (17)
C3—C4—C5—C6	0.7 (3)	O3—S1—C15—C14	11.35 (19)
C4—C5—C6—C7	−0.4 (4)	O2—S1—C15—C14	144.39 (17)
C5—C6—C7—C8	0.0 (3)	N1—S1—C15—C14	−102.03 (17)
C6—C7—C8—C3	0.1 (3)	O3—S1—C15—C10	−168.59 (16)
C6—C7—C8—N1	178.25 (19)	O2—S1—C15—C10	−35.55 (19)
C4—C3—C8—C7	0.3 (3)	N1—S1—C15—C10	78.03 (18)
C2—C3—C8—C7	179.11 (18)		

Hydrogen-bond geometry (Å, º)

*Cg*2 is the centroid of the C3–C8 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···O3	0.93	2.44	3.014 (3)	120
C9—H9···O2	0.93	2.34	2.869 (3)	116
C4—H4···O1^{i}	0.93	2.51	3.343 (3)	150
C12—H12···*Cg*2^{ii}	0.93	2.71	3.638 (3)	174

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