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2-Methyl-3-phenylsulfinyl-1-benzofuran

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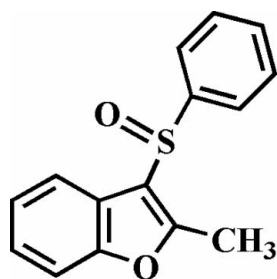
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 Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.039; wR factor = 0.103; data-to-parameter ratio = 14.7.

The title compound, $\text{C}_{15}\text{H}_{12}\text{O}_2\text{S}$, was prepared by the oxidation of 2-methyl-3-phenylsulfanyl-1-benzofuran with 3-chloroperoxybenzoic acid. The O atom and the phenyl group of the phenylsulfinyl substituent lie on opposite sides of the plane of the benzofuran system. The phenyl ring makes a dihedral angle of $78.76(4)^\circ$ with the benzofuran mean plane. The crystal structure is stabilized by π - π interactions between the furan and benzene rings of neighbouring molecules [centroid-centroid distance = $4.017(3)$ Å]. In addition, the crystal structure exhibits intermolecular $\text{C}-\text{H}\cdots\pi$ and $\text{C}-\text{H}\cdots\text{O}$ interactions.

Related literature

 For the crystal structures of similar substituted benzofuran compounds, see: Choi *et al.* (2007, 2008).


Experimental

Crystal data

$\text{C}_{15}\text{H}_{12}\text{O}_2\text{S}$	$V = 1224.6(3)$ Å ³
$M_r = 256.31$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 12.946(2)$ Å	$\mu = 0.25$ mm ⁻¹
$b = 9.466(1)$ Å	$T = 173(2)$ K
$c = 10.294(1)$ Å	$0.60 \times 0.40 \times 0.30$ mm
$\beta = 103.887(2)^\circ$	

Data collection

Bruker SMART CCD diffractometer	2405 independent reflections
Absorption correction: none	2192 reflections with $I > 2\sigma(I)$
6763 measured reflections	$R_{\text{int}} = 0.038$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$	164 parameters
$wR(F^2) = 0.102$	H-atom parameters constrained
$S = 1.11$	$\Delta\rho_{\text{max}} = 0.25$ e Å ⁻³
2405 reflections	$\Delta\rho_{\text{min}} = -0.52$ e Å ⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C4}-\text{H4}\cdots\text{O2}^{\text{i}}$	0.95	2.59	3.387(2)	142
$\text{C15}-\text{H15C}\cdots\text{O2}^{\text{ii}}$	0.98	2.42	3.232(2)	141
$\text{C12}-\text{H12}\cdots\text{Cg2}^{\text{iii}}$	0.95	3.20	3.629(3)	152

 Symmetry codes: (i) $x, -y + \frac{1}{2}, z + \frac{1}{2}$; (ii) $x, -y + \frac{3}{2}, z + \frac{1}{2}$; (iii) $-x, -y + 1, -z + 1$. Cg2 is the centroid of the C2-C7 benzene ring.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2156).

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supporting information

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2-Methyl-3-phenylsulfinyl-1-benzofuran

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S1. Comment

This work is related to our communications on the synthesis and structures of 3-phenylsulfinyl-1-benzofuran analogues, *viz.* 2,5-dimethyl-3-phenylsulfinyl-1-benzofuran (Choi *et al.*, 2007) and 2,4,6,7-tetramethyl-3-phenylsulfinyl-1-benzofuran (Choi *et al.*, 2008). Here we report the crystal structure of the title compound, 2-methyl-3-phenylsulfinyl-1-benzofuran (Fig. 1).

The benzofuran unit is almost planar, with a mean deviation of 0.008 (1) ° from the least-squares plane defined by the nine constituent atoms. The phenyl ring (C9—C14) is almost perpendicular to the plane of the benzofuran ring system [78.76 (4)°] and is tilted slightly towards it. The crystal packing (Fig. 2) is stabilized by aromatic π - π interactions between the furan and the benzene rings of neighbouring molecules. The Cg1...Cg2^{iv} distance is 4.017 (3) Å (Cg1 and Cg2 are the centroids of the C1/C2/C7/O1/C8 furan ring and the C2—C7 benzene ring, respectively, symmetry code as in Fig. 2). The crystal packing is further stabilized by C—H... π interactions between a phenyl H atom of the phenylsulfinyl substituent and the benzene ring of the benzofuran unit, with a C12-H12...Cg2ⁱⁱⁱ separation of 3.20 Å (Fig. 2 and Table 1; Cg2 is the centroid of the C2-C7 benzene ring, symmetry code as in Fig. 2). Additionally, intermolecular C—H...O interactions in the structure were observed (Fig. 2 and Table 1; symmetry code as in Fig. 2).

S2. Experimental

77% 3-Chloroperoxybenzoic acid (247 mg, 1.1 mmol) was added in small portions to a stirred solution of 2-methyl-3-phenylsulfonyl-1-benzofuran (240 mg, 1.0 mmol) in dichloromethane (30 ml) at 273 K. After being stirred for 3 h at room temperature, the mixture was washed with saturated sodium bicarbonate solution and the organic layer was separated, dried over magnesium sulfate, filtered and concentrated under vacuum. The residue was purified by column chromatography (hexane-ethyl acetate, 1:2 v/v) to afford the title compound as a colorless solid [yield 78%, m.p. 417–418 K; R_f = 0.71 (hexane-ethyl acetate, 1:2 v/v)]. Single crystals suitable for X-ray diffraction were prepared by evaporation of a solution of the title compound in acetone at room temperature. Spectroscopic analysis: ¹H NMR (CDCl₃, 400 MHz) δ 2.77 (s, 3H), 7.06 (t, J = 7.80 Hz, 1H), 7.19–7.23 (m, 2H), 7.39–7.52 (m, 4H), 7.68 (d, J = 7.44 Hz, 2H); EI—MS 256 [M^+].

S3. Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95 Å for aromatic H atoms and 0.98 Å for methyl H atoms, respectively, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms.

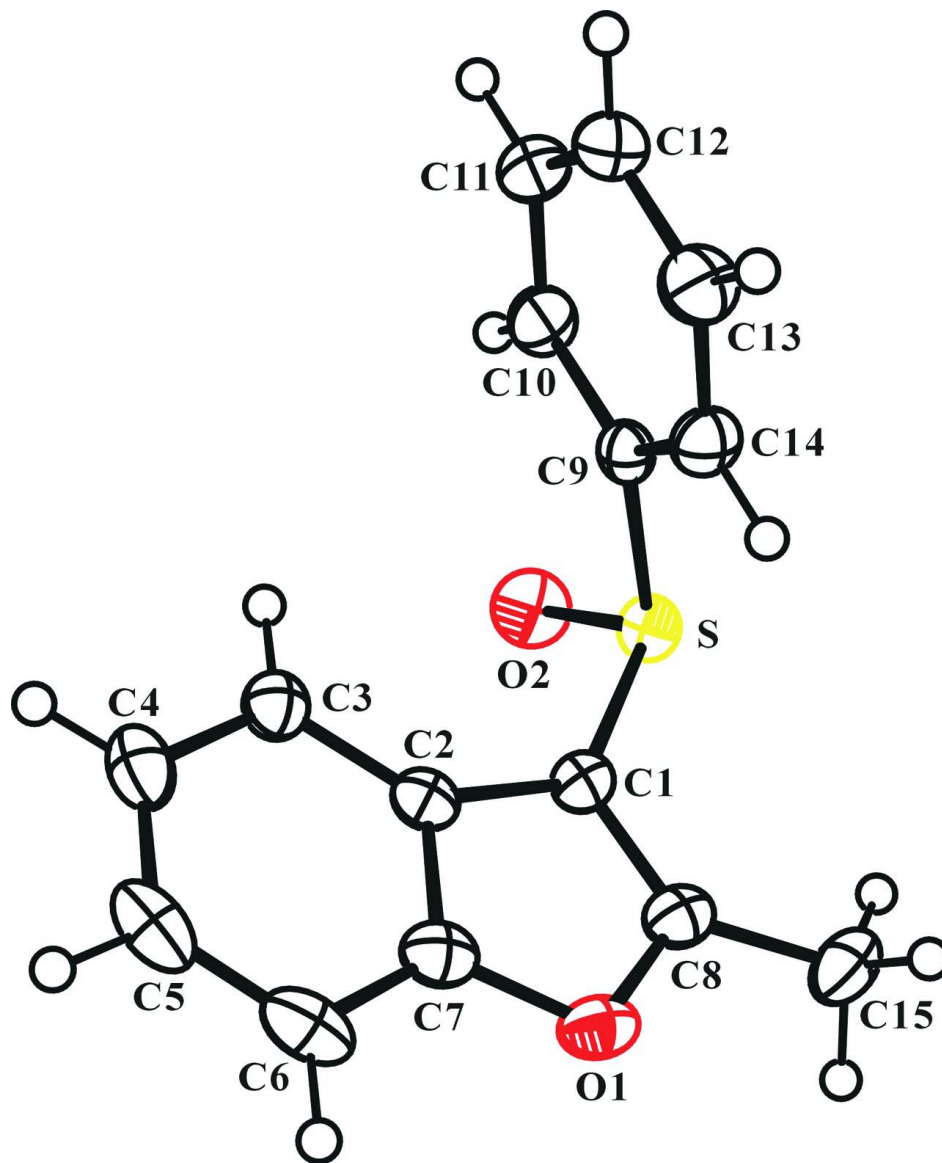


Figure 1

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

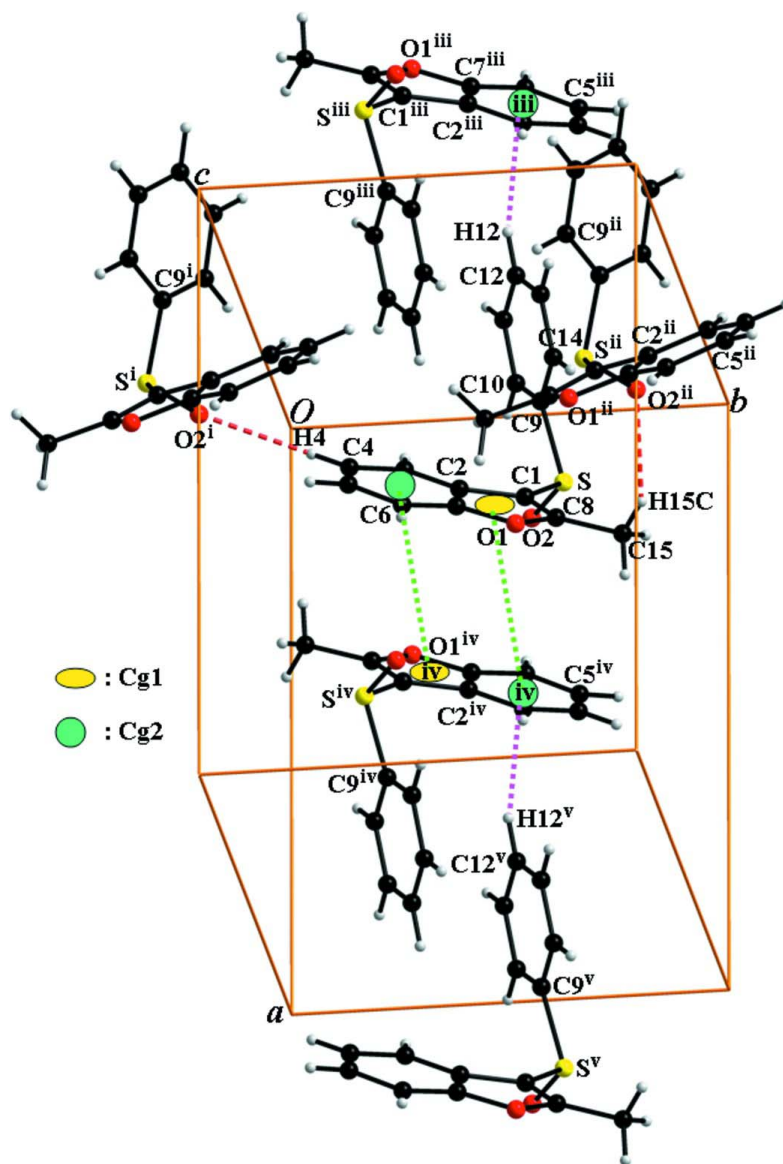


Figure 2

π - π , C—H \cdots π and C—H \cdots O interactions (dotted lines) in the title compound. Cg denotes the ring centroid. [Symmetry codes: (i) $x, -y + 1/2, z + 1/2$; (ii) $x, -y + 3/2, z + 1/2$; (iii) $-x, -y + 1, -z + 1$; (iv) $-x + 1, -y + 1, -z + 1$; (v) $x + 1, y, z$.]

2-Methyl-3-phenylsulfinyl-1-benzofuran

Crystal data

C₁₅H₁₂O₂S
M_r = 256.31
 Monoclinic, $P2_1/c$
 Hall symbol: -P 2ybc
a = 12.946 (2) Å
b = 9.466 (1) Å
c = 10.294 (1) Å
 β = 103.887 (2)°

V = 1224.6 (3) Å³
Z = 4
F(000) = 536
D_x = 1.390 Mg m⁻³
 Mo *K* α radiation, λ = 0.71073 Å
 Cell parameters from 5415 reflections
 θ = 2.7–28.2°
 μ = 0.25 mm⁻¹

$T = 173$ K $0.60 \times 0.40 \times 0.30$ mm
 Block, colorless

Data collection

Bruker SMART CCD diffractometer	2405 independent reflections
Radiation source: fine-focus sealed tube	2192 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{\text{int}} = 0.038$
Detector resolution: 10.0 pixels mm^{-1}	$\theta_{\text{max}} = 26.0^\circ$, $\theta_{\text{min}} = 1.6^\circ$
φ and ω scans	$h = -15 \rightarrow 15$
6763 measured reflections	$k = -11 \rightarrow 7$
	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.039$	H-atom parameters constrained
$wR(F^2) = 0.102$	$w = 1/[\sigma^2(F_o^2) + (0.0544P)^2 + 0.4838P]$
$S = 1.11$	where $P = (F_o^2 + 2F_c^2)/3$
2405 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
164 parameters	$\Delta\rho_{\text{max}} = 0.25 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.52 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S	0.21943 (3)	0.67373 (4)	0.24290 (4)	0.02584 (14)
O1	0.42892 (9)	0.63644 (14)	0.58590 (11)	0.0324 (3)
O2	0.23114 (10)	0.57743 (14)	0.13205 (11)	0.0372 (3)
C1	0.30363 (12)	0.61722 (17)	0.39339 (15)	0.0239 (3)
C2	0.31131 (11)	0.48127 (17)	0.45930 (14)	0.0244 (3)
C3	0.26168 (13)	0.35014 (17)	0.43207 (16)	0.0287 (3)
H3	0.2089	0.3341	0.3517	0.034*
C4	0.29212 (14)	0.24396 (19)	0.52657 (18)	0.0353 (4)
H4	0.2590	0.1540	0.5107	0.042*
C5	0.37026 (15)	0.2664 (2)	0.64428 (18)	0.0394 (4)
H5	0.3885	0.1916	0.7071	0.047*
C6	0.42179 (14)	0.3951 (2)	0.67166 (17)	0.0379 (4)
H6	0.4760	0.4104	0.7507	0.045*
C7	0.38969 (12)	0.49963 (19)	0.57741 (15)	0.0291 (4)
C8	0.37502 (12)	0.70491 (18)	0.47218 (15)	0.0277 (3)

C9	0.09419 (12)	0.62970 (16)	0.27953 (14)	0.0235 (3)
C10	0.02207 (13)	0.55290 (18)	0.18570 (16)	0.0317 (4)
H10	0.0412	0.5168	0.1086	0.038*
C11	-0.07920 (14)	0.5289 (2)	0.20534 (19)	0.0374 (4)
H11	-0.1296	0.4765	0.1412	0.045*
C12	-0.10656 (13)	0.58118 (19)	0.31772 (19)	0.0362 (4)
H12	-0.1759	0.5651	0.3306	0.043*
C13	-0.03302 (14)	0.65707 (19)	0.41178 (18)	0.0359 (4)
H13	-0.0519	0.6921	0.4894	0.043*
C14	0.06790 (13)	0.68229 (17)	0.39348 (16)	0.0295 (4)
H14	0.1183	0.7347	0.4578	0.035*
C15	0.40351 (14)	0.85446 (19)	0.45966 (19)	0.0364 (4)
H15A	0.3707	0.8880	0.3691	0.055*
H15B	0.4810	0.8633	0.4766	0.055*
H15C	0.3778	0.9113	0.5250	0.055*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0309 (2)	0.0267 (2)	0.0210 (2)	-0.00105 (15)	0.00834 (16)	0.00241 (14)
O1	0.0259 (6)	0.0415 (7)	0.0281 (6)	-0.0028 (5)	0.0033 (5)	-0.0067 (5)
O2	0.0444 (7)	0.0478 (8)	0.0227 (6)	0.0018 (6)	0.0143 (5)	-0.0049 (5)
C1	0.0239 (7)	0.0266 (8)	0.0227 (7)	-0.0017 (6)	0.0083 (6)	-0.0008 (6)
C2	0.0223 (7)	0.0298 (8)	0.0225 (7)	0.0032 (6)	0.0082 (6)	0.0006 (6)
C3	0.0291 (8)	0.0281 (8)	0.0300 (8)	0.0009 (6)	0.0094 (6)	-0.0002 (6)
C4	0.0382 (9)	0.0290 (9)	0.0431 (9)	0.0080 (7)	0.0184 (8)	0.0058 (7)
C5	0.0419 (10)	0.0439 (11)	0.0363 (9)	0.0196 (8)	0.0171 (8)	0.0148 (8)
C6	0.0333 (9)	0.0535 (11)	0.0260 (8)	0.0157 (8)	0.0056 (7)	0.0036 (8)
C7	0.0244 (7)	0.0381 (9)	0.0260 (8)	0.0029 (7)	0.0082 (6)	-0.0034 (7)
C8	0.0234 (7)	0.0340 (9)	0.0278 (8)	-0.0023 (6)	0.0103 (6)	-0.0051 (7)
C9	0.0270 (8)	0.0215 (7)	0.0211 (7)	0.0020 (6)	0.0041 (6)	0.0024 (6)
C10	0.0346 (8)	0.0323 (9)	0.0257 (8)	0.0012 (7)	0.0026 (6)	-0.0051 (7)
C11	0.0301 (9)	0.0344 (9)	0.0422 (9)	-0.0027 (7)	-0.0023 (7)	-0.0063 (8)
C12	0.0264 (8)	0.0342 (9)	0.0480 (10)	0.0003 (7)	0.0086 (7)	0.0015 (8)
C13	0.0353 (9)	0.0392 (10)	0.0358 (9)	0.0014 (7)	0.0141 (7)	-0.0051 (7)
C14	0.0303 (8)	0.0317 (9)	0.0263 (8)	-0.0020 (6)	0.0062 (6)	-0.0061 (6)
C15	0.0356 (9)	0.0336 (9)	0.0441 (10)	-0.0114 (7)	0.0175 (8)	-0.0116 (8)

Geometric parameters (Å, °)

S—O2	1.496 (1)	C6—H6	0.9500
S—C1	1.750 (2)	C8—C15	1.476 (2)
S—C9	1.800 (2)	C9—C10	1.378 (2)
O1—C8	1.373 (2)	C9—C14	1.390 (2)
O1—C7	1.386 (2)	C10—C11	1.392 (2)
C1—C8	1.357 (2)	C10—H10	0.9500
C1—C2	1.447 (2)	C11—C12	1.380 (3)
C2—C3	1.395 (2)	C11—H11	0.9500

C2—C7	1.395 (2)	C12—C13	1.385 (3)
C3—C4	1.388 (2)	C12—H12	0.9500
C3—H3	0.9500	C13—C14	1.385 (2)
C4—C5	1.396 (3)	C13—H13	0.9500
C4—H4	0.9500	C14—H14	0.9500
C5—C6	1.385 (3)	C15—H15A	0.9800
C5—H5	0.9500	C15—H15B	0.9800
C6—C7	1.378 (2)	C15—H15C	0.9800
O2—S—C1	109.46 (7)	C1—C8—C15	133.1 (2)
O2—S—C9	106.26 (7)	O1—C8—C15	116.1 (1)
C1—S—C9	98.33 (7)	C10—C9—C14	121.2 (2)
C8—O1—C7	106.5 (1)	C10—C9—S	117.9 (1)
C8—C1—C2	107.7 (1)	C14—C9—S	120.6 (1)
C8—C1—S	122.2 (1)	C9—C10—C11	119.2 (2)
C2—C1—S	130.2 (1)	C9—C10—H10	120.4
C3—C2—C7	119.2 (2)	C11—C10—H10	120.4
C3—C2—C1	136.1 (1)	C12—C11—C10	120.2 (2)
C7—C2—C1	104.6 (1)	C12—C11—H11	119.9
C4—C3—C2	117.6 (2)	C10—C11—H11	119.9
C4—C3—H3	121.2	C11—C12—C13	120.0 (2)
C2—C3—H3	121.2	C11—C12—H12	120.0
C3—C4—C5	121.5 (2)	C13—C12—H12	120.0
C3—C4—H4	119.2	C14—C13—C12	120.5 (2)
C5—C4—H4	119.2	C14—C13—H13	119.8
C6—C5—C4	121.6 (2)	C12—C13—H13	119.8
C6—C5—H5	119.2	C13—C14—C9	118.9 (2)
C4—C5—H5	119.2	C13—C14—H14	120.6
C7—C6—C5	116.1 (2)	C9—C14—H14	120.6
C7—C6—H6	122.0	C8—C15—H15A	109.5
C5—C6—H6	122.0	C8—C15—H15B	109.5
C6—C7—O1	125.6 (2)	H15A—C15—H15B	109.5
C6—C7—C2	123.9 (2)	C8—C15—H15C	109.5
O1—C7—C2	110.5 (1)	H15A—C15—H15C	109.5
C1—C8—O1	110.8 (2)	H15B—C15—H15C	109.5
O2—S—C1—C8	126.3 (1)	C1—C2—C7—O1	-0.1 (2)
C9—S—C1—C8	-123.2 (1)	C2—C1—C8—O1	-0.3 (2)
O2—S—C1—C2	-55.6 (2)	S—C1—C8—O1	178.2 (1)
C9—S—C1—C2	55.0 (2)	C2—C1—C8—C15	-179.5 (2)
C8—C1—C2—C3	-179.9 (2)	S—C1—C8—C15	-1.0 (3)
S—C1—C2—C3	1.7 (3)	C7—O1—C8—C1	0.3 (2)
C8—C1—C2—C7	0.2 (2)	C7—O1—C8—C15	179.6 (1)
S—C1—C2—C7	-178.1 (1)	O2—S—C9—C10	-17.0 (2)
C7—C2—C3—C4	1.1 (2)	C1—S—C9—C10	-130.2 (1)
C1—C2—C3—C4	-178.7 (2)	O2—S—C9—C14	168.5 (1)
C2—C3—C4—C5	-0.6 (2)	C1—S—C9—C14	55.3 (1)
C3—C4—C5—C6	-0.7 (3)	C14—C9—C10—C11	0.6 (2)

C4—C5—C6—C7	1.3 (2)	S—C9—C10—C11	-173.9 (1)
C5—C6—C7—O1	178.7 (1)	C9—C10—C11—C12	-0.2 (3)
C5—C6—C7—C2	-0.8 (2)	C10—C11—C12—C13	-0.3 (3)
C8—O1—C7—C6	-179.6 (2)	C11—C12—C13—C14	0.6 (3)
C8—O1—C7—C2	-0.1 (2)	C12—C13—C14—C9	-0.3 (3)
C3—C2—C7—C6	-0.4 (2)	C10—C9—C14—C13	-0.3 (2)
C1—C2—C7—C6	179.4 (2)	S—C9—C14—C13	174.0 (1)
C3—C2—C7—O1	-179.9 (1)		

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>
C4—H4 \cdots O2 ⁱ	0.95	2.59	3.387 (2)	142
C15—H15C \cdots O2 ⁱⁱ	0.98	2.42	3.232 (2)	141
C12—H12 \cdots Cg2 ⁱⁱⁱ	0.95	3.20	3.629 (3)	152

Symmetry codes: (i) $x, -y+1/2, z+1/2$; (ii) $x, -y+3/2, z+1/2$; (iii) $-x, -y+1, -z+1$.