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2,4,6-Trimethyl-3-phenylsulfinyl-1-benzofuran

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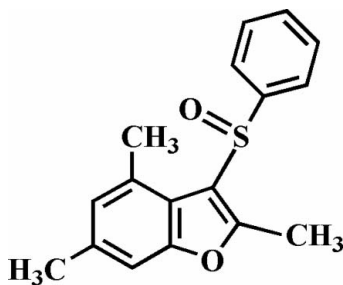
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Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.065; wR factor = 0.152; data-to-parameter ratio = 14.8.

The title compound, $\text{C}_{17}\text{H}_{16}\text{O}_2\text{S}$, was prepared by the oxidation of 2,4,6-trimethyl-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 planar benzofuran fragment. The phenyl ring is nearly perpendicular to the plane of the benzofuran unit [$89.88(8)^\circ$] and is tilted slightly towards it. The crystal structure is stabilized by $\text{C}-\text{H}\cdots\pi$ interactions between a phenyl H atoms and the phenyl and furan rings of neighbouring molecules. In addition, the crystal structure exhibits intermolecular $\text{C}-\text{H}\cdots\text{O}$ interactions.

Related literature

For the crystal structures of similar 3-phenylsulfinyl-1-benzofuran derivatives, see: Choi *et al.* (2007, 2008).



Experimental

Crystal data

$\text{C}_{17}\text{H}_{16}\text{O}_2\text{S}$
 $M_r = 284.36$
 Monoclinic, $P2_1/c$
 $a = 13.493(2)$ Å
 $b = 6.0154(8)$ Å
 $c = 17.269(2)$ Å
 $\beta = 90.909(3)^\circ$

$V = 1401.5(3)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.23$ mm⁻¹
 $T = 173(2)$ K
 $0.40 \times 0.40 \times 0.30$ mm

Data collection

Bruker SMART CCD diffractometer
 Absorption correction: none
 7482 measured reflections

2731 independent reflections
 2291 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.067$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.065$
 $wR(F^2) = 0.151$
 $S = 1.18$
 2731 reflections

184 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.76$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.43$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C12}-\text{H12}\cdots\text{Cg1}^{\text{i}}$	0.95	2.88	3.673 (5)	142
$\text{C13}-\text{H13}\cdots\text{Cg2}^{\text{ii}}$	0.95	2.95	3.882 (5)	168
$\text{C16}-\text{H16C}\cdots\text{O2}^{\text{iii}}$	0.98	2.40	3.366 (4)	167

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x, -y+1, -z+1$. Cg1 and Cg2 are the centroids of the $\text{C9}-\text{C14}$ phenyl ring and the C1/C2/C7/O1/C8 furan ring, respectively.

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: BX2157).

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supporting information

Acta Cryst. (2008). E64, o1476 [doi:10.1107/S1600536808021090]

2,4,6-Trimethyl-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, 2,4,6-trimethyl-3-phenylsulfinyl-1-benzofuran (Fig. 1).

The benzofuran unit is essentially planar, with a mean deviation of 0.013 (2) Å 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 system [89.88 (8)°] and is tilted slightly towards it. The molecular packing (Fig. 2) is stabilized by two different C—H \cdots π interactions; one between a phenyl H atom and the phenyl ring of the phenylsulfinyl substituent, with a C12—H12 \cdots Cg1ⁱ separation of 2.88 Å, and a second between a phenyl H atom and the furan ring of the benzofuran unit, with a C13—H13 \cdots Cg2ⁱⁱ separation of 2.95 Å (Fig. 2 and Table 1; Cg1 and Cg2 are the centroids of the C9—C14 phenyl ring and the C1/C2/C7/O1/C8 furan ring, respectively, symmetry code as in Fig. 2). Additionally, intermolecular C—H \cdots 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 2,4,6-trimethyl-3-phenylsulfinyl-1-benzofuran (268 mg, 1.0 mmol) in dichloromethane (20 ml) at 273 K. After being stirred at room temperature for 3 h, 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, 1:1 *v/v*) to afford the title compound as a colorless solid [yield 79%, m.p. 404–405 K; R_f = 0.68 (hexane-ethyl acetate, 1:1 *v/v*)]. Single crystals suitable for X-ray diffraction were prepared by slow evaporation of a solution of the title compound in acetone at room temperature. Spectroscopic analysis: ¹H NMR (CDCl₃, 400 MHz) δ 2.17 (s, 3H), 2.37 (s, 3H), 2.69 (s, 3H), 6.77 (s, 1H), 7.09 (s, 1H), 7.40–7.47 (m, 3H), 7.48–7.52 (m, 2H); EI—MS 284 [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.

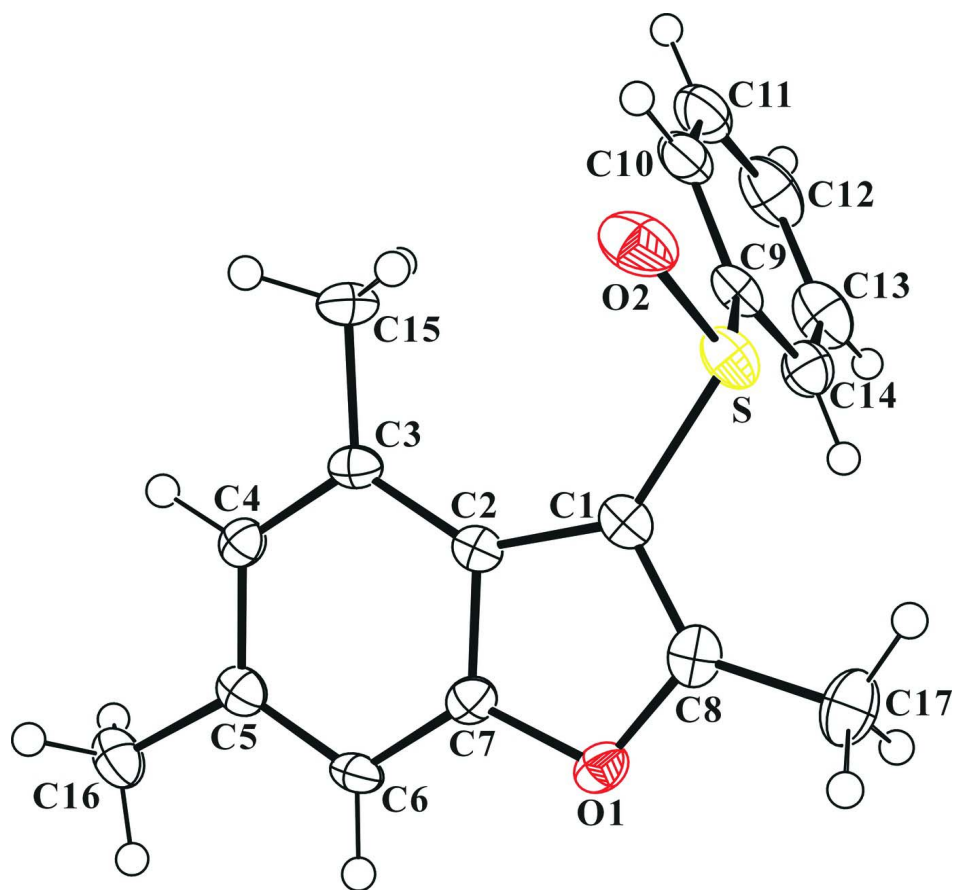


Figure 1

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

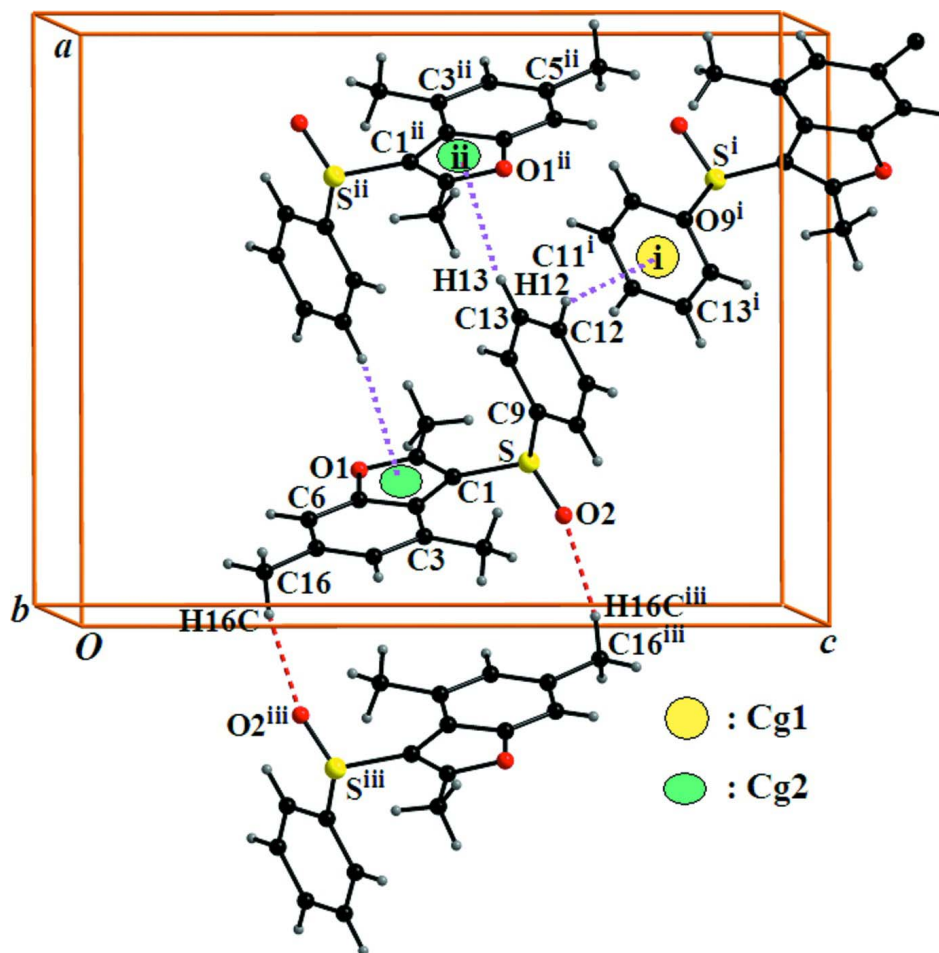


Figure 2

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

2,4,6-Trimethyl-3-phenylsulfinyl-1-benzofuran

Crystal data

$C_{17}H_{16}O_2S$

$M_r = 284.36$

Monoclinic, $P2_1/c$

Hall symbol: $-P_2ybc$

$a = 13.493 (2) \text{ \AA}$

$b = 6.0154 (8) \text{ \AA}$

$c = 17.269 (2) \text{ \AA}$

$\beta = 90.909 (3)^\circ$

$V = 1401.5 (3) \text{ \AA}^3$

$Z = 4$

$F(000) = 600$

$D_x = 1.348 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3769 reflections

$\theta = 2.4\text{--}28.3^\circ$

$\mu = 0.23 \text{ mm}^{-1}$

$T = 173 \text{ K}$

Block, colorless

$0.40 \times 0.40 \times 0.30 \text{ mm}$

Data collection

Bruker SMART CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: $10.0 \text{ pixels mm}^{-1}$

φ and ω scans

7482 measured reflections

2731 independent reflections

2291 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.067$
 $\theta_{\text{max}} = 26.0^\circ$, $\theta_{\text{min}} = 1.5^\circ$

$h = -16 \rightarrow 13$
 $k = -7 \rightarrow 7$
 $l = -21 \rightarrow 21$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.065$
 $wR(F^2) = 0.151$
 $S = 1.18$
 2731 reflections
 184 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
 $w = 1/[\sigma^2(F_o^2) + (0.044P)^2 + 2.2792P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.76 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.43 \text{ e } \text{\AA}^{-3}$

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S	0.27007 (6)	0.17129 (13)	0.60901 (4)	0.0260 (2)
O1	0.25645 (15)	0.1919 (3)	0.38328 (11)	0.0233 (5)
O2	0.17960 (18)	0.1863 (4)	0.65645 (13)	0.0381 (6)
C1	0.2432 (2)	0.2412 (5)	0.51168 (16)	0.0207 (6)
C2	0.1875 (2)	0.4202 (5)	0.47392 (15)	0.0187 (6)
C3	0.1297 (2)	0.6036 (5)	0.49618 (16)	0.0195 (6)
C4	0.0910 (2)	0.7354 (5)	0.43702 (16)	0.0205 (6)
H4	0.0517	0.8599	0.4508	0.025*
C5	0.1066 (2)	0.6950 (5)	0.35773 (16)	0.0220 (6)
C6	0.1615 (2)	0.5103 (5)	0.33600 (15)	0.0210 (6)
H6	0.1720	0.4754	0.2831	0.025*
C7	0.1998 (2)	0.3805 (5)	0.39487 (16)	0.0199 (6)
C8	0.2813 (2)	0.1107 (5)	0.45543 (18)	0.0228 (6)
C9	0.3464 (2)	0.4068 (5)	0.63557 (16)	0.0235 (7)
C10	0.3202 (2)	0.5406 (6)	0.69695 (17)	0.0310 (8)
H10	0.2594	0.5171	0.7227	0.037*
C11	0.3838 (3)	0.7098 (6)	0.7205 (2)	0.0384 (9)
H11	0.3653	0.8069	0.7612	0.046*
C12	0.4737 (3)	0.7374 (6)	0.6850 (2)	0.0415 (9)
H12	0.5176	0.8516	0.7019	0.050*
C13	0.5001 (3)	0.5990 (6)	0.6247 (2)	0.0361 (8)
H13	0.5624	0.6179	0.6006	0.043*

C14	0.4362 (2)	0.4331 (6)	0.59928 (18)	0.0278 (7)
H14	0.4539	0.3389	0.5576	0.033*
C15	0.1085 (2)	0.6565 (6)	0.57950 (16)	0.0269 (7)
H15A	0.1651	0.7359	0.6026	0.040*
H15B	0.0974	0.5181	0.6080	0.040*
H15C	0.0492	0.7500	0.5821	0.040*
C16	0.0615 (2)	0.8484 (6)	0.29790 (18)	0.0300 (7)
H16A	0.0769	0.7938	0.2460	0.045*
H16B	0.0889	0.9982	0.3047	0.045*
H16C	-0.0105	0.8530	0.3041	0.045*
C17	0.3438 (3)	-0.0921 (6)	0.4550 (2)	0.0342 (8)
H17A	0.3529	-0.1465	0.5081	0.051*
H17B	0.4084	-0.0567	0.4331	0.051*
H17C	0.3112	-0.2070	0.4235	0.051*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0282 (4)	0.0237 (4)	0.0258 (4)	-0.0067 (3)	-0.0038 (3)	0.0092 (3)
O1	0.0217 (10)	0.0217 (11)	0.0265 (11)	-0.0011 (9)	0.0024 (8)	-0.0063 (9)
O2	0.0366 (13)	0.0508 (16)	0.0271 (12)	-0.0123 (12)	0.0029 (10)	0.0099 (11)
C1	0.0198 (14)	0.0202 (15)	0.0221 (14)	-0.0066 (12)	-0.0014 (11)	0.0027 (11)
C2	0.0188 (14)	0.0197 (15)	0.0176 (13)	-0.0042 (12)	0.0001 (11)	0.0010 (11)
C3	0.0194 (14)	0.0218 (15)	0.0174 (14)	-0.0064 (12)	0.0015 (11)	-0.0033 (11)
C4	0.0179 (14)	0.0205 (15)	0.0232 (15)	-0.0012 (12)	0.0013 (11)	-0.0014 (11)
C5	0.0194 (14)	0.0253 (16)	0.0212 (14)	-0.0043 (12)	-0.0005 (11)	0.0019 (12)
C6	0.0216 (15)	0.0283 (16)	0.0130 (13)	-0.0039 (13)	-0.0002 (11)	-0.0012 (12)
C7	0.0184 (14)	0.0182 (15)	0.0231 (14)	-0.0036 (11)	0.0001 (11)	-0.0049 (11)
C8	0.0192 (14)	0.0164 (15)	0.0329 (17)	-0.0061 (11)	0.0003 (12)	0.0008 (12)
C9	0.0267 (16)	0.0241 (16)	0.0193 (14)	-0.0033 (13)	-0.0073 (12)	0.0055 (12)
C10	0.0309 (17)	0.041 (2)	0.0210 (15)	0.0025 (15)	-0.0058 (13)	0.0047 (14)
C11	0.050 (2)	0.035 (2)	0.0296 (18)	0.0064 (17)	-0.0146 (16)	-0.0049 (15)
C12	0.047 (2)	0.033 (2)	0.044 (2)	-0.0130 (17)	-0.0219 (17)	0.0051 (16)
C13	0.0270 (17)	0.041 (2)	0.0399 (19)	-0.0118 (15)	-0.0070 (14)	0.0100 (16)
C14	0.0233 (16)	0.0304 (17)	0.0297 (16)	-0.0012 (14)	-0.0025 (13)	0.0007 (14)
C15	0.0338 (17)	0.0292 (17)	0.0179 (14)	-0.0005 (14)	0.0023 (12)	-0.0054 (13)
C16	0.0267 (16)	0.0355 (19)	0.0277 (16)	-0.0005 (14)	-0.0034 (13)	0.0086 (14)
C17	0.0304 (18)	0.0227 (17)	0.050 (2)	0.0015 (14)	0.0054 (15)	0.0032 (15)

Geometric parameters (Å, °)

S—O2	1.484 (2)	C9—C14	1.382 (4)
S—C1	1.765 (3)	C10—C11	1.388 (5)
S—C9	1.806 (3)	C10—H10	0.9500
O1—C8	1.375 (4)	C11—C12	1.378 (6)
O1—C7	1.385 (4)	C11—H11	0.9500
C1—C8	1.356 (4)	C12—C13	1.384 (5)
C1—C2	1.461 (4)	C12—H12	0.9500

C2—C7	1.398 (4)	C13—C14	1.385 (5)
C2—C3	1.407 (4)	C13—H13	0.9500
C3—C4	1.389 (4)	C14—H14	0.9500
C3—C15	1.506 (4)	C15—H15A	0.9800
C4—C5	1.410 (4)	C15—H15B	0.9800
C4—H4	0.9500	C15—H15C	0.9800
C5—C6	1.390 (4)	C16—H16A	0.9800
C5—C16	1.506 (4)	C16—H16B	0.9800
C6—C7	1.376 (4)	C16—H16C	0.9800
C6—H6	0.9500	C17—H17A	0.9800
C8—C17	1.483 (4)	C17—H17B	0.9800
C9—C10	1.382 (5)	C17—H17C	0.9800
O2—S—C1	110.69 (14)	C9—C10—H10	120.4
O2—S—C9	106.44 (15)	C11—C10—H10	120.4
C1—S—C9	99.35 (13)	C12—C11—C10	120.1 (3)
C8—O1—C7	106.7 (2)	C12—C11—H11	119.9
C8—C1—C2	107.8 (2)	C10—C11—H11	119.9
C8—C1—S	118.0 (2)	C11—C12—C13	120.1 (3)
C2—C1—S	134.2 (2)	C11—C12—H12	119.9
C7—C2—C3	118.4 (3)	C13—C12—H12	119.9
C7—C2—C1	104.0 (2)	C12—C13—C14	120.3 (3)
C3—C2—C1	137.6 (3)	C12—C13—H13	119.8
C4—C3—C2	116.7 (3)	C14—C13—H13	119.8
C4—C3—C15	120.5 (3)	C9—C14—C13	119.0 (3)
C2—C3—C15	122.8 (3)	C9—C14—H14	120.5
C3—C4—C5	123.8 (3)	C13—C14—H14	120.5
C3—C4—H4	118.1	C3—C15—H15A	109.5
C5—C4—H4	118.1	C3—C15—H15B	109.5
C6—C5—C4	119.2 (3)	H15A—C15—H15B	109.5
C6—C5—C16	121.0 (3)	C3—C15—H15C	109.5
C4—C5—C16	119.7 (3)	H15A—C15—H15C	109.5
C7—C6—C5	116.7 (3)	H15B—C15—H15C	109.5
C7—C6—H6	121.6	C5—C16—H16A	109.5
C5—C6—H6	121.6	C5—C16—H16B	109.5
C6—C7—O1	124.0 (3)	H16A—C16—H16B	109.5
C6—C7—C2	125.1 (3)	C5—C16—H16C	109.5
O1—C7—C2	110.8 (2)	H16A—C16—H16C	109.5
C1—C8—O1	110.7 (3)	H16B—C16—H16C	109.5
C1—C8—C17	134.5 (3)	C8—C17—H17A	109.5
O1—C8—C17	114.7 (3)	C8—C17—H17B	109.5
C10—C9—C14	121.2 (3)	H17A—C17—H17B	109.5
C10—C9—S	120.0 (2)	C8—C17—H17C	109.5
C14—C9—S	118.4 (2)	H17A—C17—H17C	109.5
C9—C10—C11	119.2 (3)	H17B—C17—H17C	109.5
O2—S—C1—C8	-135.5 (2)	C3—C2—C7—C6	1.5 (4)
C9—S—C1—C8	112.9 (2)	C1—C2—C7—C6	-178.8 (3)

O2—S—C1—C2	45.8 (3)	C3—C2—C7—O1	-178.6 (2)
C9—S—C1—C2	-65.8 (3)	C1—C2—C7—O1	1.0 (3)
C8—C1—C2—C7	-1.3 (3)	C2—C1—C8—O1	1.1 (3)
S—C1—C2—C7	177.5 (2)	S—C1—C8—O1	-177.90 (18)
C8—C1—C2—C3	178.3 (3)	C2—C1—C8—C17	179.6 (3)
S—C1—C2—C3	-2.9 (5)	S—C1—C8—C17	0.6 (5)
C7—C2—C3—C4	-1.5 (4)	C7—O1—C8—C1	-0.5 (3)
C1—C2—C3—C4	179.0 (3)	C7—O1—C8—C17	-179.3 (2)
C7—C2—C3—C15	177.6 (3)	O2—S—C9—C10	7.6 (3)
C1—C2—C3—C15	-1.9 (5)	C1—S—C9—C10	122.5 (3)
C2—C3—C4—C5	-0.1 (4)	O2—S—C9—C14	-179.8 (2)
C15—C3—C4—C5	-179.3 (3)	C1—S—C9—C14	-64.9 (3)
C3—C4—C5—C6	1.8 (4)	C14—C9—C10—C11	2.5 (5)
C3—C4—C5—C16	-179.9 (3)	S—C9—C10—C11	174.8 (2)
C4—C5—C6—C7	-1.8 (4)	C9—C10—C11—C12	-2.6 (5)
C16—C5—C6—C7	179.9 (3)	C10—C11—C12—C13	1.2 (5)
C5—C6—C7—O1	-179.6 (3)	C11—C12—C13—C14	0.5 (5)
C5—C6—C7—C2	0.2 (4)	C10—C9—C14—C13	-0.8 (5)
C8—O1—C7—C6	179.4 (3)	S—C9—C14—C13	-173.3 (2)
C8—O1—C7—C2	-0.4 (3)	C12—C13—C14—C9	-0.7 (5)

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...Cg1 ⁱ	0.95	2.88	3.673 (5)	142
C13—H13...Cg2 ⁱⁱ	0.95	2.95	3.882 (5)	168
C16—H16C...O2 ⁱⁱⁱ	0.98	2.40	3.366 (4)	167

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