Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,5-Dimethyl-3-phenylsulfinyl-1-benzofuran

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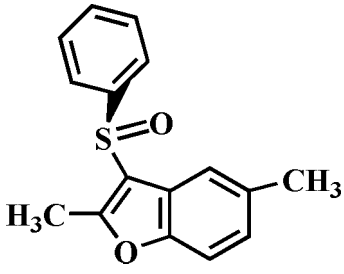
Received 30 August 2007; accepted 7 September 2007

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.050; wR factor = 0.118; data-to-parameter ratio = 16.5.

The title compound, $C_{16}H_{14}O_2S$, was prepared by the oxidation of 2,5-dimethyl-3-phenylsulfanyl-1-benzofuran using 3-chloroperbenzoic 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 is almost perpendicular to the plane of the benzofuran unit [87.72 (6)°] and is tilted slightly towards it. The crystal structure is stabilized by intermolecular C—H$\cdots\pi$ interactions between the 5-methyl group and the furan ring of the benzofuran system, and C—H$\cdots$O=S hydrogen bonds.

Related literature

For the crystal structures of isomers of the title compound, see: Choi *et al.* (2007); Seo *et al.* (2007).



Experimental

Crystal data

$C_{16}H_{14}O_2S$
M_r = 270.33
Monoclinic, $P2_1/c$
a = 11.4663 (6) Å
b = 6.2309 (3) Å
c = 18.847 (1) Å
β = 100.343 (1)°
V = 1324.65 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 173 (2) K
0.40 × 0.30 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
7756 measured reflections
2877 independent reflections
2365 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.118
S = 1.13
2877 reflections
174 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

Cg is the centroid of the furan ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C16—H16$A\cdots Cg^{i}$	0.98	2.75	3.640 (3)	151
C14—H14$\cdots$O2^{ii}	0.95	2.54	3.145 (3)	122
C15—H15$C\cdots$O2^{iii}	0.98	2.60	3.537 (3)	160

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); 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: CI2457).

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  doi:10.1107/S1600536807043954

supplementary materials

Acta Cryst. (2007). E**63**, o4042 [**doi:10.1107/S1600536807043954**]

2,5-Dimethyl-3-phenylsulfinyl-1-benzofuran

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Comment

As part of our continuing studies on the synthesis and structure of 2-methyl-3-phenysulfinyl-1-benzofuran analogues, we have recently described the crystal structures of 5-iodo-2-methyl-3-phenylsulfinyl-1-benzofuran (Choi *et al.*, 2007) and 5-bromo-2-methyl-3-phenylsulfinyl-1-benzofuran (Seo *et al.*, 2007). Here we report the molecular and crystal structure of the title compound, 2,5-dimethyl-3-phenylsulfinyl-1-benzofuran (Fig. 1).

The benzofuran ring system is essentially planar, with a mean deviation of 0.008 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 ring system [87.72 (6) °] and is tilted slightly towards it. The molecular packing (Fig. 2) is stabilized by intermolecular C—H···π interactions between a hydrogen of the 5-methyl group and the furan ring of the benzofuran system (Table 1, *Cg* is the centroid of the O1/C2/C1/C8/C3 furan ring), forming a chain along the *b* axis (Fig. 2). Further stability comes from weak C—H···O hydrogen bonds (Table 1).

Experimental

3-Chloroperbenzoic acid (77%, 314 mg, 1.40 mmol) was added in small portions to a stirred solution of 2,5-dimethyl-3-phenylsulfanyl-1-benzofuran (330 mg, 1.30 mmol) in dichloromethane (30 ml) at 273 K. After being stirred at room temperature for 2 h, the mixture was washed with saturated sodium bicarbonate solution and the organic layer was separated, dried over magnesium sulfate, filtered and concentrated in vacuum. The residue was purified by column chromatography (hexane-ethyl acetate, 1:1 *v*/*v*) to afford the title compound as a colourless solid [yield 83%, m.p. 406–407 K; R_f = 0.69 (hexane-ethyl acetate, 1:1 *v*/*v*)]. Single crystals suitable for X-ray diffraction were obtained by slow evaporation of a dilute solution of the title compound in chloroform at room temperature.

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, and with U_{iso}(H) = 1.2U_{eq}(C) for aromatic H atoms and U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms.

Figures

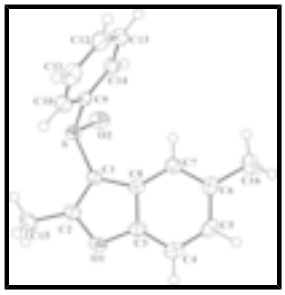


Fig. 1. The molecular structure of the title compound, showing displacement ellipsoides drawn at the 50% probability level.

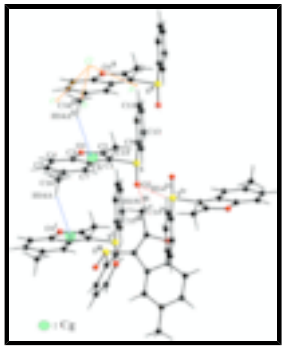


Fig. 2. The C—H···π interaction (blue dotted lines) and C—H···O hydrogen bonds (red dotted lines) in the title compound. [Symmetry code: (i) *x*, 1 + *y*, *z*; (ii) *x*, *y* − 1, *z*; (iii) 1 − *x*, *y* + 1/2, 1/2 − *z*; (iv) 1 − *x*, 2 − *y*, 1/2 − *z*.]

2,5-Dimethyl-3-phenylsulfinyl-1-benzofuran

Crystal data

$C_{16}H_{14}O_2S$	F_{000} = 568
M_r = 270.33	D_x = 1.355 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 406-407 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 11.4663 (6) Å	Cell parameters from 3453 reflections
b = 6.2309 (3) Å	θ = 2.6–28.0º
c = 18.847 (1) Å	μ = 0.24 mm^{-1}
β = 100.343 (1)º	*T* = 173 (2) K
V = 1324.65 (12) Å^3	Plate, colourless
Z = 4	0.40 × 0.30 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer	2877 independent reflections
Radiation source: fine-focus sealed tube	2365 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.040
Detector resolution: 10.0 pixels mm^{-1}	θ_{max} = 27.0º
T = 173(2) K	θ_{min} = 1.8º
φ and ω scans	*h* = −14→14
Absorption correction: none	*k* = −7→7

7756 measured reflections

$l = -23 \rightarrow 15$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.050$

$wR(F^2) = 0.118$

$S = 1.13$

2877 reflections

174 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.0467P)^2 + 0.8059P]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.43$ e Å^{-3}

$\Delta\rho_{min} = -0.32$ e Å^{-3}

Extinction correction: none

Special details

Experimental. Spectroscopic analysis: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.22 (s, 3H), 2.71 (s, 3H), 6.97–7.01 (m, 2H), 7.23 (d, J = 2.92 Hz, 1H), 7.39–7.49 (m, 3H), 7.62–7.66 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 13.11, 21.23, 110.66, 118.29, 119.87, 123.78, 124.55, 126.04, 129.09, 130.35, 133.35, 142.97, 152.55, 159.76; EI—MS 270 [M^+].

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 $F^2 > 2$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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S	0.43404 (5)	0.66525 (9)	0.09835 (3)	0.02556 (16)
O1	0.19127 (14)	0.5264 (3)	0.21547 (8)	0.0288 (4)
O2	0.47042 (14)	0.8958 (3)	0.10329 (9)	0.0357 (4)
C1	0.30788 (18)	0.6429 (3)	0.13955 (11)	0.0231 (4)
C2	0.29523 (19)	0.4935 (4)	0.18964 (11)	0.0261 (5)
C3	0.13892 (19)	0.7081 (3)	0.18066 (11)	0.0251 (5)
C4	0.0339 (2)	0.8019 (4)	0.19060 (13)	0.0326 (5)
H4	−0.0121	0.7444	0.2233	0.039*
C5	−0.0008 (2)	0.9845 (4)	0.15038 (13)	0.0316 (5)
H5	−0.0722	1.0540	0.1562	0.038*
C6	0.0654 (2)	1.0711 (4)	0.10119 (12)	0.0282 (5)
C7	0.17043 (19)	0.9709 (4)	0.09248 (12)	0.0262 (5)
H7	0.2164	1.0272	0.0596	0.031*

C8	0.20766 (18)	0.7869 (3)	0.13254 (11)	0.0222 (4)
C9	0.36209 (17)	0.6185 (3)	0.00667 (11)	0.0220 (4)
C10	0.3169 (2)	0.4165 (4)	−0.01264 (13)	0.0299 (5)
H10	0.3219	0.3054	0.0223	0.036*
C11	0.2644 (2)	0.3788 (4)	−0.08342 (14)	0.0370 (6)
H11	0.2313	0.2419	−0.0971	0.044*
C12	0.2599 (2)	0.5390 (4)	−0.13439 (13)	0.0370 (6)
H12	0.2228	0.5128	−0.1829	0.044*
C13	0.3091 (2)	0.7374 (4)	−0.11498 (13)	0.0347 (6)
H13	0.3078	0.8461	−0.1505	0.042*
C14	0.36065 (19)	0.7789 (4)	−0.04388 (13)	0.0279 (5)
H14	0.3943	0.9154	−0.0303	0.033*
C15	0.3687 (2)	0.3092 (4)	0.22060 (13)	0.0363 (6)
H15A	0.4390	0.2990	0.1979	0.054*
H15B	0.3224	0.1767	0.2117	0.054*
H15C	0.3933	0.3301	0.2727	0.054*
C16	0.0237 (2)	1.2711 (4)	0.05914 (14)	0.0373 (6)
H16A	0.0505	1.3979	0.0883	0.056*
H16B	−0.0631	1.2709	0.0471	0.056*
H16C	0.0566	1.2745	0.0146	0.056*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0192 (3)	0.0303 (3)	0.0272 (3)	−0.0016 (2)	0.0041 (2)	0.0018 (2)
O1	0.0338 (9)	0.0320 (9)	0.0221 (8)	−0.0026 (7)	0.0087 (7)	0.0035 (6)
O2	0.0326 (9)	0.0370 (10)	0.0379 (9)	−0.0148 (7)	0.0075 (7)	−0.0042 (8)
C1	0.0230 (10)	0.0236 (11)	0.0227 (10)	−0.0023 (8)	0.0038 (8)	0.0004 (9)
C2	0.0289 (11)	0.0289 (12)	0.0198 (10)	−0.0031 (9)	0.0022 (8)	−0.0015 (9)
C3	0.0276 (11)	0.0267 (11)	0.0210 (10)	−0.0038 (9)	0.0044 (8)	−0.0036 (9)
C4	0.0296 (12)	0.0416 (14)	0.0285 (12)	−0.0051 (11)	0.0101 (9)	−0.0023 (10)
C5	0.0255 (11)	0.0362 (13)	0.0332 (13)	0.0005 (10)	0.0056 (9)	−0.0106 (10)
C6	0.0289 (11)	0.0251 (11)	0.0293 (12)	−0.0013 (9)	0.0014 (9)	−0.0082 (9)
C7	0.0253 (11)	0.0248 (11)	0.0287 (11)	−0.0045 (9)	0.0053 (9)	0.0009 (9)
C8	0.0217 (10)	0.0227 (11)	0.0224 (10)	−0.0046 (8)	0.0046 (8)	−0.0045 (8)
C9	0.0182 (9)	0.0237 (11)	0.0249 (10)	0.0004 (8)	0.0057 (8)	−0.0009 (8)
C10	0.0308 (12)	0.0251 (12)	0.0353 (13)	−0.0031 (9)	0.0102 (10)	0.0022 (10)
C11	0.0313 (12)	0.0332 (13)	0.0463 (15)	−0.0040 (10)	0.0062 (11)	−0.0117 (11)
C12	0.0282 (12)	0.0526 (16)	0.0288 (12)	0.0083 (11)	0.0012 (10)	−0.0077 (11)
C13	0.0330 (12)	0.0411 (14)	0.0307 (12)	0.0091 (11)	0.0070 (10)	0.0086 (11)
C14	0.0250 (11)	0.0246 (11)	0.0348 (12)	0.0007 (9)	0.0074 (9)	0.0043 (9)
C15	0.0444 (14)	0.0353 (14)	0.0277 (12)	0.0036 (11)	0.0022 (10)	0.0078 (10)
C16	0.0354 (13)	0.0289 (13)	0.0456 (15)	0.0040 (10)	0.0017 (11)	−0.0005 (11)

Geometric parameters (Å, °)

S—O2	1.494 (2)	C9—C14	1.379 (3)
S—C1	1.766 (2)	C9—C10	1.385 (3)
S—C9	1.800 (2)	C10—C11	1.381 (3)

O1—C2	1.381 (3)	C10—H10	0.95
O1—C3	1.390 (3)	C11—C12	1.380 (4)
C1—C2	1.352 (3)	C11—H11	0.95
C1—C8	1.445 (3)	C12—C13	1.381 (4)
C2—C15	1.481 (3)	C12—H12	0.95
C3—C4	1.381 (3)	C13—C14	1.388 (3)
C3—C8	1.393 (3)	C13—H13	0.95
C4—C5	1.385 (3)	C14—H14	0.95
C4—H4	0.95	C15—H15A	0.98
C5—C6	1.407 (3)	C15—H15B	0.98
C5—H5	0.95	C15—H15C	0.98
C6—C7	1.392 (3)	C16—H16A	0.98
C6—C16	1.508 (3)	C16—H16B	0.98
C7—C8	1.397 (3)	C16—H16C	0.98
C7—H7	0.95		
O2—S—C1	106.86 (9)	C14—C9—S	119.5 (2)
O2—S—C9	106.9 (1)	C10—C9—S	119.0 (2)
C1—S—C9	97.98 (9)	C11—C10—C9	119.1 (2)
C2—O1—C3	106.3 (2)	C11—C10—H10	120.5
C2—C1—C8	107.9 (2)	C9—C10—H10	120.5
C2—C1—S	124.1 (2)	C12—C11—C10	120.3 (2)
C8—C1—S	127.7 (2)	C12—C11—H11	119.8
C1—C2—O1	110.6 (2)	C10—C11—H11	119.8
C1—C2—C15	133.2 (2)	C11—C12—C13	120.0 (2)
O1—C2—C15	116.2 (2)	C11—C12—H12	120.0
C4—C3—O1	126.4 (2)	C13—C12—H12	120.0
C4—C3—C8	123.2 (2)	C12—C13—C14	120.4 (2)
O1—C3—C8	110.3 (2)	C12—C13—H13	119.8
C3—C4—C5	116.4 (2)	C14—C13—H13	119.8
C3—C4—H4	121.8	C9—C14—C13	118.8 (2)
C5—C4—H4	121.8	C9—C14—H14	120.6
C4—C5—C6	122.7 (2)	C13—C14—H14	120.6
C4—C5—H5	118.7	C2—C15—H15A	109.5
C6—C5—H5	118.7	C2—C15—H15B	109.5
C7—C6—C5	119.1 (2)	H15A—C15—H15B	109.5
C7—C6—C16	120.5 (2)	C2—C15—H15C	109.5
C5—C6—C16	120.4 (2)	H15A—C15—H15C	109.5
C6—C7—C8	119.4 (2)	H15B—C15—H15C	109.5
C6—C7—H7	120.3	C6—C16—H16A	109.5
C8—C7—H7	120.3	C6—C16—H16B	109.5
C3—C8—C7	119.2 (2)	H16A—C16—H16B	109.5
C3—C8—C1	104.8 (2)	C6—C16—H16C	109.5
C7—C8—C1	136.0 (2)	H16A—C16—H16C	109.5
C14—C9—C10	121.3 (2)	H16B—C16—H16C	109.5
O2—S—C1—C2	−131.0 (2)	C4—C3—C8—C1	179.8 (2)
C9—S—C1—C2	118.6 (2)	O1—C3—C8—C1	−0.3 (2)
O2—S—C1—C8	42.3 (2)	C6—C7—C8—C3	−0.3 (3)
C9—S—C1—C8	−68.1 (2)	C6—C7—C8—C1	−179.1 (2)

C8—C1—C2—O1	1.2 (2)	C2—C1—C8—C3	−0.6 (2)
S—C1—C2—O1	175.6 (1)	S—C1—C8—C3	−174.7 (2)
C8—C1—C2—C15	−178.8 (2)	C2—C1—C8—C7	178.4 (2)
S—C1—C2—C15	−4.4 (4)	S—C1—C8—C7	4.3 (4)
C3—O1—C2—C1	−1.4 (2)	O2—S—C9—C14	6.0 (2)
C3—O1—C2—C15	178.6 (2)	C1—S—C9—C14	116.4 (2)
C2—O1—C3—C4	−179.1 (2)	O2—S—C9—C10	−178.7 (2)
C2—O1—C3—C8	1.1 (2)	C1—S—C9—C10	−68.3 (2)
O1—C3—C4—C5	179.4 (2)	C14—C9—C10—C11	−3.1 (3)
C8—C3—C4—C5	−0.8 (3)	S—C9—C10—C11	−178.3 (2)
C3—C4—C5—C6	0.6 (3)	C9—C10—C11—C12	1.6 (4)
C4—C5—C6—C7	−0.3 (3)	C10—C11—C12—C13	0.8 (4)
C4—C5—C6—C16	−179.6 (2)	C11—C12—C13—C14	−1.7 (4)
C5—C6—C7—C8	0.1 (3)	C10—C9—C14—C13	2.2 (3)
C16—C6—C7—C8	179.4 (2)	S—C9—C14—C13	177.4 (2)
C4—C3—C8—C7	0.7 (3)	C12—C13—C14—C9	0.3 (3)
O1—C3—C8—C7	−179.5 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16A···Cg^{i}	0.98	2.75	3.640 (3)	151
C14—H14···O2^{ii}	0.95	2.54	3.145 (3)	122
C15—H15C···O2^{iii}	0.98	2.60	3.537 (3)	160

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

Fig. 1

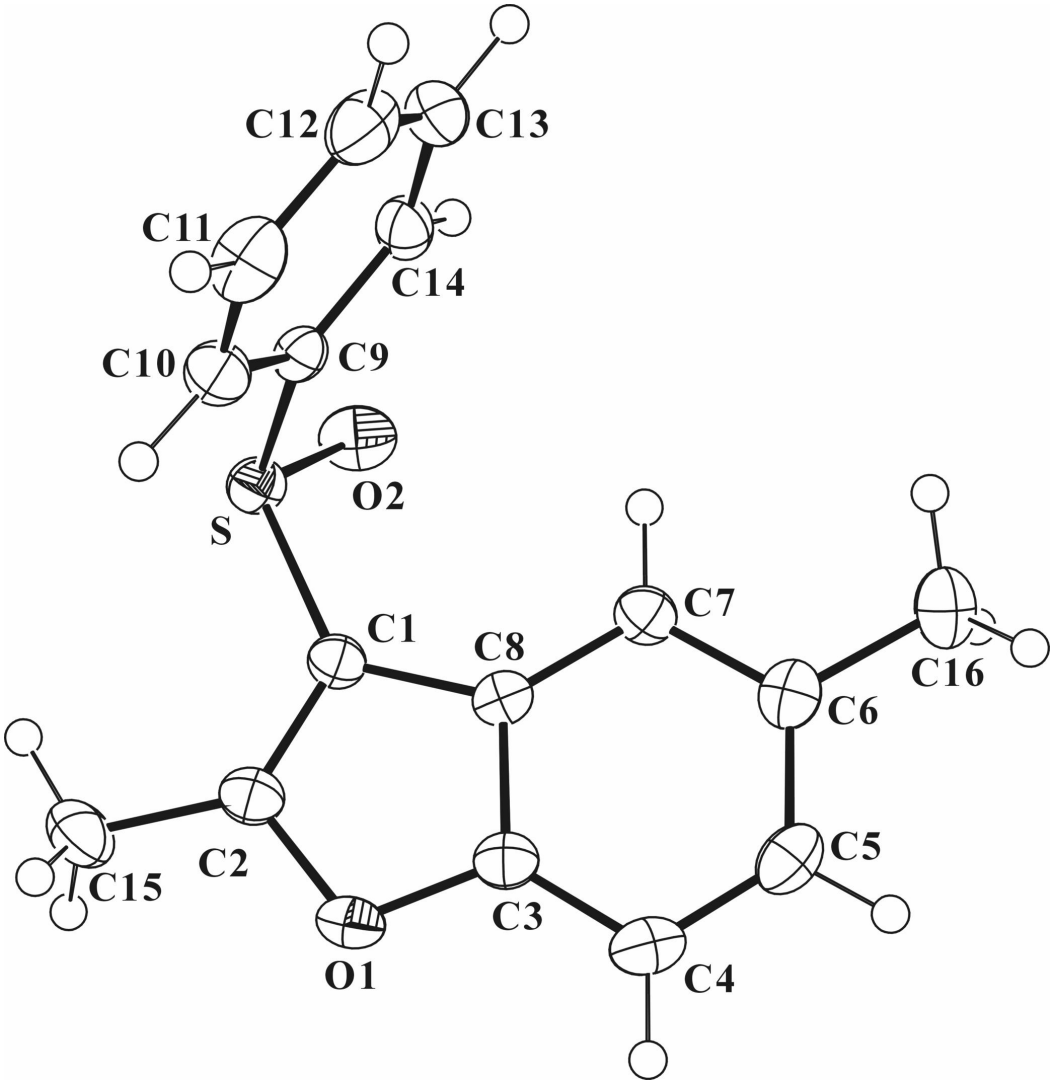
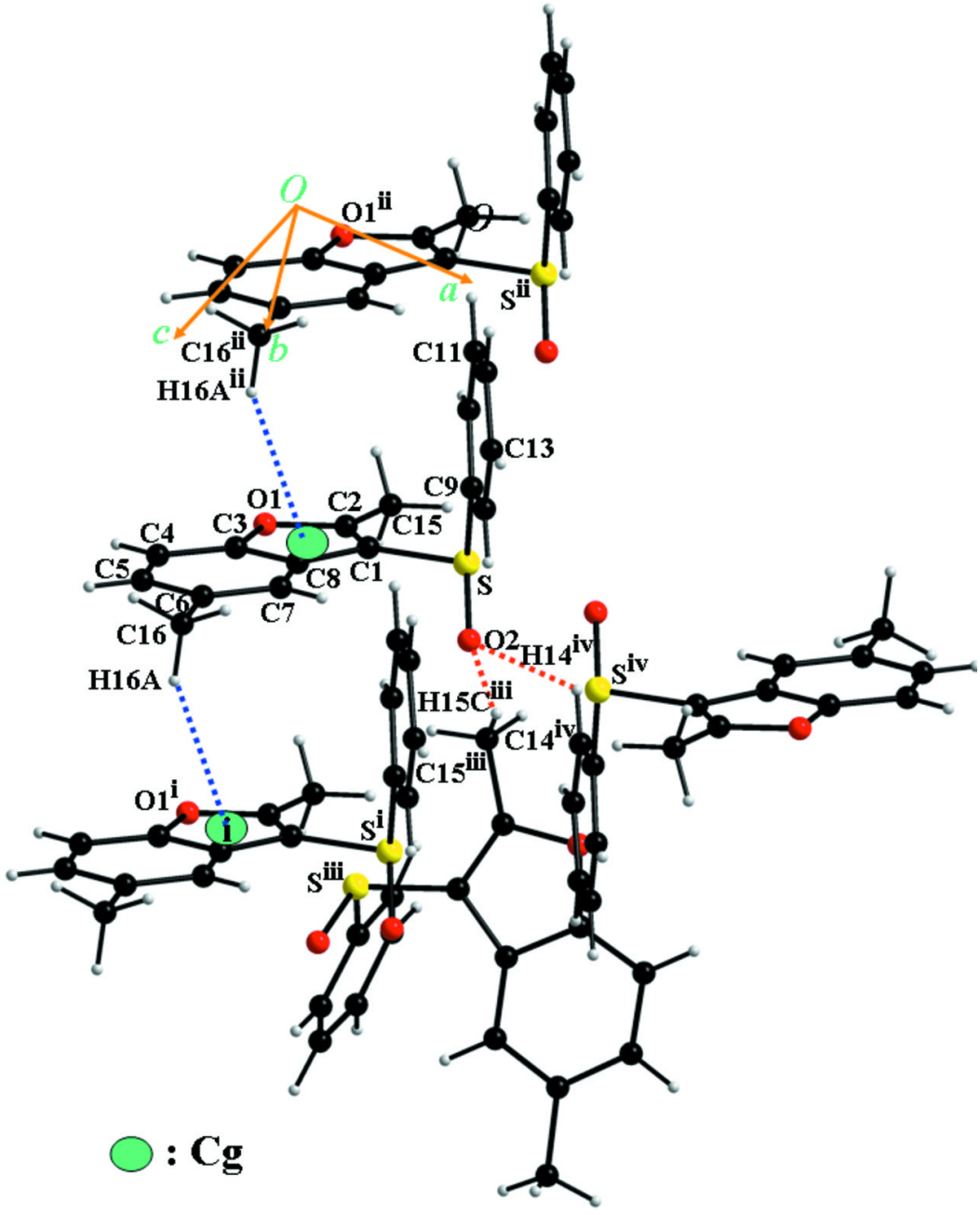


Fig. 2


O
O1ii
a
Sii
c
C16ii
b
H16Aii
C11
C13
C9
O1
C2
C3
C15
C4
C5
C8
C1
S
C6
C7
C16
O2
H14iv
Siv
H16A
H15Ciii
C14iv
C15iii
O1i
i
Si
Siii
: Cg