Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-Methyl-3-methylsulfinyl-2-phenyl-1-benzofuran

Hong Dae Choi,[a] Pil Ja Seo,[a] Byeng Wha Son[b] and Uk Lee[b]*

[a]Department of Chemistry, Dongeui University, San 24 Kaya-dong Busanjin-gu, Busan 614-714, Republic of Korea, and [b]Department of Chemistry, Pukyong National University, 599-1 Daeyeon 3-dong Nam-gu, Busan 608-737, Republic of Korea
Correspondence e-mail: uklee@pknu.ac.kr

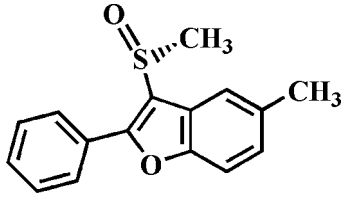
Received 14 May 2007; accepted 16 May 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.114; data-to-parameter ratio = 15.4.

The title compound, $C_{16}H_{14}O_2S$, was prepared by the oxidation of 3,5-dimethyl-2-phenyl-1-benzofuran using 3-chloroperbenzoic acid. The O atom and the methyl group of the methylsulfinyl substituent lie on opposite sides of the plane of the benzofuran fragment. The phenyl ring is rotated out of the benzofuran plane, with a dihedral angle of 31.94 (7)°. The crystal packing is stabilized by C—H···π(phenyl ring) interactions.

Related literature

For the crystal structures of isomers of the title compound, see: Choi *et al.* (2007*a*,*b*). For details of the pharmacological properties of benzofuran compounds, see: Howlett *et al.* (1999) and Ward (1997).



Experimental

Crystal data

$C_{16}H_{14}O_2S$
M_r = 270.33
Triclinic, $P\overline{1}$
a = 5.3788 (5) Å
b = 8.7492 (8) Å
c = 15.120 (1) Å
α = 78.140 (2)°
β = 85.231 (2)°
γ = 83.248 (2)°
V = 690.26 (10) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 298 (2) K
0.52 × 0.41 × 0.23 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: none
3934 measured reflections
2673 independent reflections
2308 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.114
S = 1.05
2673 reflections
174 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

Cg is the centroid of the phenyl ring.

D—H···A	D—H	H···A	D···A	D—H···A
C16—H16B···Cg^{i}	0.96	3.14	3.941 (2)	142

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

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: *ORTEPIII* (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: CV2245).

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  doi:10.1107/S1600536807024269

supplementary materials

Acta Cryst. (2007). E**63**, o2922 [**doi:10.1107/S1600536807024269**]

5-Methyl-3-methylsulfinyl-2-phenyl-1-benzofuran

H. D. Choi, P. J. Seo, B. W. Son and U. Lee

Comment

1-Benzofuran ring systems have attracted considerable interest because of their various pharmacological properties (Howlett *et al.*, 1999; Ward, 1997). With our continuing studies on the synthesis and structures of 2-phenyl-benzofuran derivatives, the crystal structures of 5-chloro-3-methylsulfinyl-2-phenyl-1-benzofuran (Choi *et al.*, 2007*a*) and 5-bromo-3-methylsulfinyl-2-phenyl-1-benzofuran (Choi *et al.*, 2007*b*) have been described to the literature. Herein we report the molecular and crystal structure of the title compound (I) (Fig. 1).

The benzofuran unit is essentially planar, with a mean deviation of 0.016 Å from the least-squares plane defined by the nine constituent atoms. The dihedral angle in (I) formed by the plane of the benzofuran ring and the plane of phenyl ring is 31.94 (7)°. The molecular packing (Fig. 2) is stabilized by CH_2—H···π interactions between the *S*-methyl group and a phenyl ring, with a C16—H16B···*Cg*^{i} separation of 3.14 Å (*Cg* is a centroid of the C9—C14 phenyl ring, symmetry code as in Fig. 2).

Experimental

3-Chloroperbenzoic acid (77%, 471 mg, 2.1 mmol) was added in small portions to a stirred solution of 3,5-dimethyl-2-phenyl-1-benzofuran (508 mg, 2.0 mmol) in dichloromethane (30 ml) at 273 K. After being stirred at room temperature for 1 h, the mixture was washed with saturated sodium bicarbonate solution and the organic layer separated, dried over magnesium sulfate, filtered and concentrated in vacuum. The residue was purified by column chromatography (ethyl acetate) to afford the title compound as a colorless solid [yield 83%, m.p. 417–418 K; R_f = 0.56 (ethyl acetate)]. Single crystals suitable for X-ray diffraction were prepared by slow evaporation of a dilute solution of title compound (I) in tetrahydrofuran at room temperature.

Refinement

All H atoms were geometrically located in ideal positions and refined using a riding model, with C—H = 0.93 Å for aromatic H atoms and C—H = 0.96 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

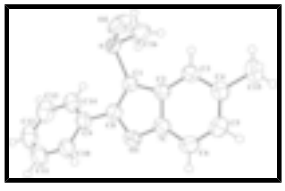


Fig. 1. The molecular structure of (I), showing displacement ellipsoids drawn at the 50% probability level.

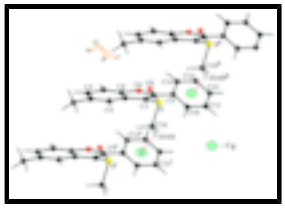


Fig. 2. CH_2—H···π interactions in (I). *Cg* denotes ring centroid. [Symmetry codes: (i) 1 + *x*, *y*, *z*; (ii) *x* − 1, *y*, *z*.]

5-Methyl-3-methylsulfinyl-2-phenyl-1-benzofuran

Crystal data

$C_{16}H_{14}O_2S$	$Z = 2$
$M_r = 270.33$	$F_{000} = 284$
Triclinic, $P\bar{1}$	$D_x = 1.301$ Mg m^{-3}
Hall symbol: -p_1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 5.3788$ (5) Å	Cell parameters from 2704 reflections
$b = 8.7492$ (8) Å	$\theta = 2.4$–28.2°
$c = 15.1200$ (10) Å	$\mu = 0.23$ mm^{-1}
$\alpha = 78.140$ (2)°	$T = 298$ (2) K
$\beta = 85.231$ (2)°	Block, colourless
$\gamma = 83.248$ (2)°	0.52 × 0.41 × 0.23 mm
$V = 690.26$ (10) Å^3	

Data collection

Bruker SMART CCD diffractometer	2673 independent reflections
Radiation source: fine-focus sealed tube	2308 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.026$
Detector resolution: 10.00 pixels mm^{-1}	$\theta_{max} = 26.0^\circ$
$T = 298$(2) K	$\theta_{min} = 2.4^\circ$
φ and ω scans	$h = -6 \rightarrow 6$
Absorption correction: none	$k = -10 \rightarrow 9$
3934 measured reflections	$l = -17 \rightarrow 18$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.040$	H-atom parameters constrained
$wR(F^2) = 0.114$	$w = 1/[\sigma^2(F_o^2) + (0.0633P)^2 + 0.1495P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
2673 reflections	$\Delta\rho_{max} = 0.25$ e Å^{-3}
174 parameters	$\Delta\rho_{min} = -0.23$ e Å^{-3}

Primary atom site location: structure-invariant direct methods

Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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	−0.01847 (8)	0.17113 (5)	0.39289 (3)	0.04671 (17)
O1	−0.0475 (2)	0.41996 (15)	0.14345 (9)	0.0543 (3)
O2	−0.0081 (3)	0.00196 (16)	0.39122 (10)	0.0737 (5)
C1	0.0325 (3)	0.2664 (2)	0.27927 (12)	0.0437 (4)
C2	0.2146 (3)	0.2144 (2)	0.21257 (11)	0.0442 (4)
C3	0.4194 (3)	0.1002 (2)	0.21359 (13)	0.0490 (4)
H3	0.4613	0.0326	0.2674	0.059*
C4	0.5596 (3)	0.0887 (2)	0.13358 (13)	0.0539 (5)
C5	0.4902 (4)	0.1901 (3)	0.05283 (14)	0.0652 (6)
H5	0.5848	0.1812	−0.0006	0.078*
C6	0.2868 (4)	0.3026 (3)	0.04971 (13)	0.0647 (6)
H6	0.2412	0.3685	−0.0042	0.078*
C7	0.1552 (3)	0.3118 (2)	0.13069 (12)	0.0502 (4)
C8	−0.1166 (3)	0.3897 (2)	0.23446 (12)	0.0472 (4)
C9	−0.3267 (3)	0.4955 (2)	0.26260 (13)	0.0494 (4)
C10	−0.3412 (4)	0.5378 (3)	0.34680 (16)	0.0616 (5)
H10	−0.2165	0.4981	0.3869	0.074*
C11	−0.5421 (4)	0.6394 (3)	0.37099 (18)	0.0740 (7)
H11	−0.5535	0.6656	0.4279	0.089*
C12	−0.7245 (4)	0.7014 (3)	0.3110 (2)	0.0752 (7)
H12	−0.8571	0.7707	0.3271	0.090*
C13	−0.7103 (4)	0.6609 (3)	0.22781 (18)	0.0705 (6)
H13	−0.8349	0.7018	0.1878	0.085*
C14	−0.5121 (3)	0.5594 (2)	0.20282 (15)	0.0575 (5)
H14	−0.5028	0.5339	0.1458	0.069*
C15	0.7849 (4)	−0.0321 (3)	0.13411 (17)	0.0691 (6)
H15A	0.8440	−0.0616	0.1940	0.104*
H15B	0.9155	0.0116	0.0928	0.104*
H15C	0.7382	−0.1231	0.1158	0.104*
C16	0.2699 (4)	0.2019 (3)	0.43396 (15)	0.0777 (7)
H16A	0.2749	0.1545	0.4970	0.117*

H16B	0.2818	0.3126	0.4262	0.117*
H16C	0.4079	0.1553	0.4007	0.117*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0475 (3)	0.0457 (3)	0.0452 (3)	−0.00823 (18)	0.00619 (18)	−0.00651 (18)
O1	0.0574 (8)	0.0514 (7)	0.0472 (7)	0.0105 (6)	−0.0044 (6)	−0.0024 (6)
O2	0.1148 (13)	0.0462 (8)	0.0576 (9)	−0.0207 (8)	0.0182 (8)	−0.0072 (6)
C1	0.0433 (9)	0.0421 (9)	0.0440 (9)	−0.0032 (7)	−0.0001 (7)	−0.0064 (7)
C2	0.0460 (9)	0.0417 (9)	0.0436 (9)	−0.0033 (7)	−0.0012 (7)	−0.0065 (7)
C3	0.0483 (10)	0.0440 (9)	0.0519 (10)	0.0002 (7)	−0.0046 (8)	−0.0053 (8)
C4	0.0493 (10)	0.0529 (11)	0.0593 (11)	0.0006 (8)	−0.0006 (8)	−0.0148 (9)
C5	0.0680 (13)	0.0746 (14)	0.0498 (11)	0.0048 (10)	0.0072 (9)	−0.0162 (10)
C6	0.0766 (14)	0.0673 (13)	0.0431 (10)	0.0089 (10)	−0.0031 (9)	−0.0040 (9)
C7	0.0515 (10)	0.0479 (10)	0.0477 (10)	0.0057 (8)	−0.0044 (8)	−0.0071 (8)
C8	0.0472 (9)	0.0441 (9)	0.0485 (10)	−0.0026 (7)	−0.0015 (7)	−0.0072 (7)
C9	0.0445 (9)	0.0388 (9)	0.0631 (11)	−0.0032 (7)	0.0017 (8)	−0.0085 (8)
C10	0.0588 (12)	0.0562 (11)	0.0717 (13)	−0.0003 (9)	−0.0027 (9)	−0.0207 (10)
C11	0.0749 (15)	0.0616 (13)	0.0912 (17)	−0.0074 (11)	0.0137 (13)	−0.0356 (13)
C12	0.0557 (12)	0.0493 (12)	0.119 (2)	0.0022 (9)	0.0129 (13)	−0.0245 (13)
C13	0.0519 (11)	0.0546 (12)	0.0989 (18)	0.0056 (9)	−0.0028 (11)	−0.0080 (12)
C14	0.0477 (10)	0.0495 (10)	0.0716 (13)	−0.0007 (8)	−0.0020 (9)	−0.0067 (9)
C15	0.0563 (12)	0.0690 (14)	0.0811 (15)	0.0095 (10)	0.0013 (10)	−0.0240 (11)
C16	0.0682 (14)	0.1050 (19)	0.0569 (13)	−0.0326 (13)	−0.0148 (10)	0.0087 (12)

Geometric parameters (Å, °)

S—O2	1.4799 (14)	C9—C10	1.391 (3)
S—C1	1.7630 (17)	C9—C14	1.391 (3)
S—C16	1.787 (2)	C10—C11	1.391 (3)
O1—C8	1.375 (2)	C10—H10	0.9300
O1—C7	1.385 (2)	C11—C12	1.378 (3)
C1—C8	1.361 (2)	C11—H11	0.9300
C1—C2	1.450 (2)	C12—C13	1.370 (4)
C2—C7	1.389 (2)	C12—H12	0.9300
C2—C3	1.396 (2)	C13—C14	1.384 (3)
C3—C4	1.387 (3)	C13—H13	0.9300
C3—H3	0.9300	C14—H14	0.9300
C4—C5	1.404 (3)	C15—H15A	0.9600
C4—C15	1.511 (3)	C15—H15B	0.9600
C5—C6	1.380 (3)	C15—H15C	0.9600
C5—H5	0.9300	C16—H16A	0.9600
C6—C7	1.375 (3)	C16—H16B	0.9600
C6—H6	0.9300	C16—H16C	0.9600
C8—C9	1.467 (2)		
O2—S—C1	105.74 (8)	C10—C9—C8	121.54 (17)
O2—S—C16	107.32 (12)	C14—C9—C8	119.48 (18)

C1—S—C16	98.26 (9)	C9—C10—C11	120.0 (2)
C8—O1—C7	106.44 (13)	C9—C10—H10	120.0
C8—C1—C2	106.90 (15)	C11—C10—H10	120.0
C8—C1—S	125.75 (14)	C12—C11—C10	120.3 (2)
C2—C1—S	126.70 (13)	C12—C11—H11	119.9
C7—C2—C3	118.70 (16)	C10—C11—H11	119.9
C7—C2—C1	105.21 (15)	C13—C12—C11	119.9 (2)
C3—C2—C1	136.07 (16)	C13—C12—H12	120.0
C4—C3—C2	119.38 (17)	C11—C12—H12	120.0
C4—C3—H3	120.3	C12—C13—C14	120.5 (2)
C2—C3—H3	120.3	C12—C13—H13	119.8
C3—C4—C5	119.35 (18)	C14—C13—H13	119.8
C3—C4—C15	119.91 (18)	C13—C14—C9	120.3 (2)
C5—C4—C15	120.75 (18)	C13—C14—H14	119.8
C6—C5—C4	122.46 (19)	C9—C14—H14	119.8
C6—C5—H5	118.8	C4—C15—H15A	109.5
C4—C5—H5	118.8	C4—C15—H15B	109.5
C7—C6—C5	116.35 (18)	H15A—C15—H15B	109.5
C7—C6—H6	121.8	C4—C15—H15C	109.5
C5—C6—H6	121.8	H15A—C15—H15C	109.5
C6—C7—O1	125.81 (17)	H15B—C15—H15C	109.5
C6—C7—C2	123.75 (18)	S—C16—H16A	109.5
O1—C7—C2	110.42 (16)	S—C16—H16B	109.5
C1—C8—O1	111.02 (15)	H16A—C16—H16B	109.5
C1—C8—C9	134.00 (17)	S—C16—H16C	109.5
O1—C8—C9	114.97 (15)	H16A—C16—H16C	109.5
C10—C9—C14	118.95 (18)	H16B—C16—H16C	109.5
O2—S—C1—C8	127.57 (17)	C3—C2—C7—O1	−178.22 (15)
C16—S—C1—C8	−121.74 (18)	C1—C2—C7—O1	0.7 (2)
O2—S—C1—C2	−42.01 (18)	C2—C1—C8—O1	1.0 (2)
C16—S—C1—C2	68.69 (19)	S—C1—C8—O1	−170.29 (12)
C8—C1—C2—C7	−1.0 (2)	C2—C1—C8—C9	−177.78 (19)
S—C1—C2—C7	170.14 (14)	S—C1—C8—C9	10.9 (3)
C8—C1—C2—C3	177.6 (2)	C7—O1—C8—C1	−0.5 (2)
S—C1—C2—C3	−11.2 (3)	C7—O1—C8—C9	178.49 (15)
C7—C2—C3—C4	1.0 (3)	C1—C8—C9—C10	32.1 (3)
C1—C2—C3—C4	−177.57 (19)	O1—C8—C9—C10	−146.66 (18)
C2—C3—C4—C5	−1.2 (3)	C1—C8—C9—C14	−150.1 (2)
C2—C3—C4—C15	178.73 (17)	O1—C8—C9—C14	31.2 (2)
C3—C4—C5—C6	0.3 (3)	C14—C9—C10—C11	1.6 (3)
C15—C4—C5—C6	−179.6 (2)	C8—C9—C10—C11	179.47 (19)
C4—C5—C6—C7	0.7 (4)	C9—C10—C11—C12	−1.5 (3)
C5—C6—C7—O1	177.1 (2)	C10—C11—C12—C13	1.1 (4)
C5—C6—C7—C2	−0.9 (3)	C11—C12—C13—C14	−0.8 (4)
C8—O1—C7—C6	−178.4 (2)	C12—C13—C14—C9	1.0 (3)
C8—O1—C7—C2	−0.2 (2)	C10—C9—C14—C13	−1.4 (3)
C3—C2—C7—C6	0.1 (3)	C8—C9—C14—C13	−179.28 (18)
C1—C2—C7—C6	179.04 (19)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16B···Cg(phenyl)^{i}	0.96	3.14	3.941 (2)	142

Symmetry codes: (i) *x*+1, *y*, *z*.

Fig. 1

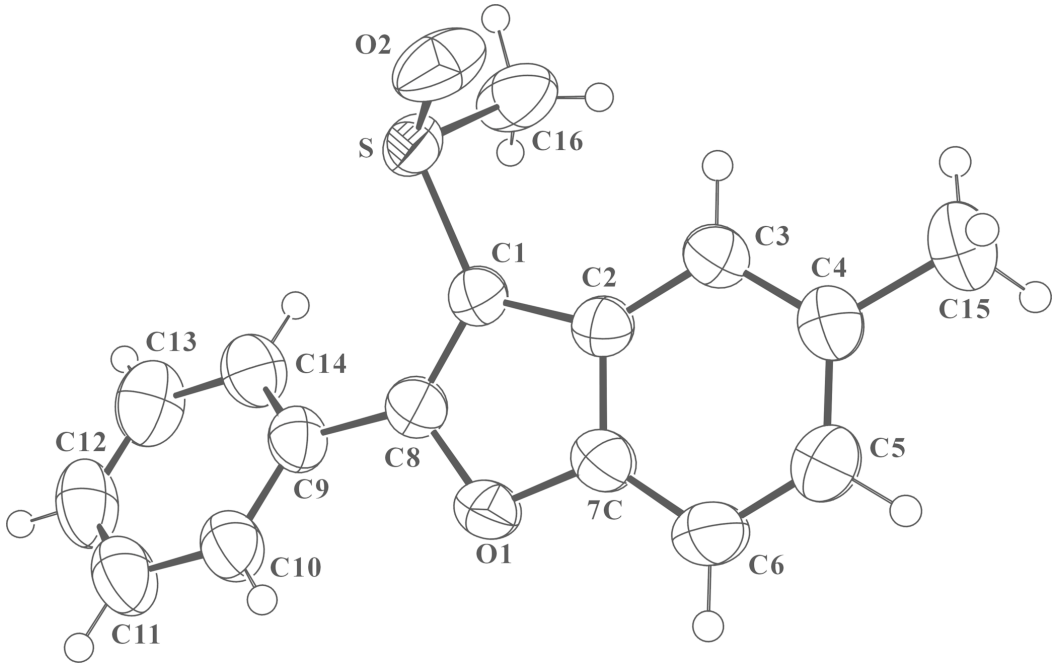
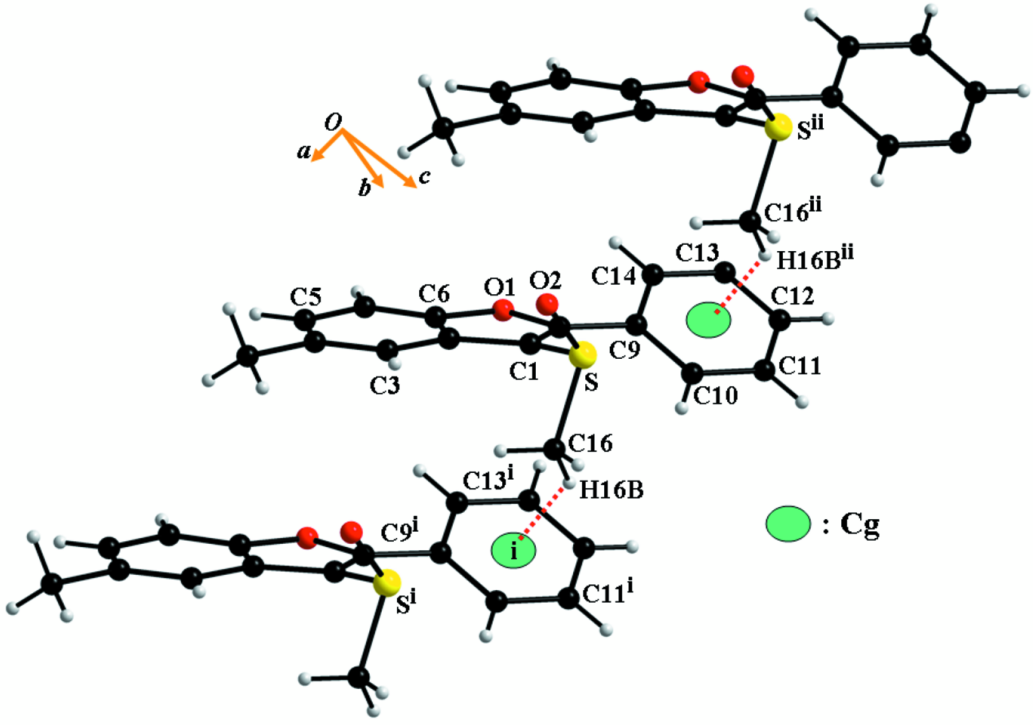


Fig. 2


O
a
b
c
S^ii
C16^ii
H16B^ii
C13
C14
C12
C5
C6
O1
O2
C9
C11
C3
C1
S
C10
C16
C13^i
H16B
C9^i
i
: Cg
S^i
C11^i