Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-Iodo-2-methyl-3-phenylsulfinyl-1-benzofuran

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

[a]Department of Chemistry, Dongeui University, San 24 Kaya-dong Busanjin-gu, Busan 614-714, Republic of Korea, [b]Department of Chemistry, Dongeui University, San 24 Kaya-dong Busanjin-gu, Busan 614-714, Republic of Korea, and [c]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

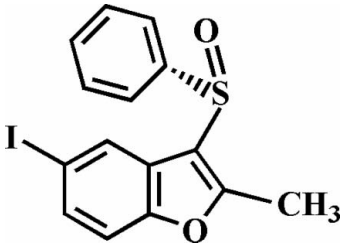
Received 13 August 2007; accepted 15 August 2007

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.004 Å; R factor = 0.025; wR factor = 0.066; data-to-parameter ratio = 17.6.

The title compound, $C_{15}H_{11}IO_2S$, was prepared by the oxidation of 5-iodo-2-methyl-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 [85.75 (7)°] and is tilted slightly towards it. The crystal structure is stabilized by a C—H···O hydrogen bond, and an I···O halogen bond of 3.165 (2) Å and a nearly linear C—I···O angle of 165.55 (7)°.

Related literature

For the crystal structure of an isomer of the title compound, see: Choi *et al.* (2007). For related literature, see: Allen (2002).



Experimental

Crystal data

$C_{15}H_{11}IO_2S$
M_r = 382.20
Monoclinic, $P2_1/c$
a = 12.9995 (6) Å
b = 11.5063 (5) Å
c = 9.7556 (4) Å
β = 106.004 (1)°
V = 1402.65 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.43 mm^{-1}
T = 173 (2) K
0.60 × 0.60 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1999)
T_{min} = 0.248, T_{max} = 0.613
8261 measured reflections
3049 independent reflections
2850 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.066
S = 1.05
3049 reflections
173 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.92 e Å^{-3}
$\Delta\rho_{min}$ = −1.04 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9B···O2^{i}	0.98	2.48	3.257 (3)	136

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

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: WN2192).

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Brandenburg, K. (1998). *DIAMOND*. Version 2.1. Crystal Impact GbR, Bonn, Germany.
Bruker (1997). *SMART* (Version 5.631) and *SAINT* (Version 6.12). Bruker AXS Inc., Madison, Wisconsin, USA.
Choi, H. D., Seo, P. J., Son, B. W. & Lee, U. (2007). *Acta Cryst.* E**63**, o3745.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1999). *SADABS*. University of Göttingen, Germany.

 doi:10.1107/S1600536807040482

supplementary materials

Acta Cryst. (2007). E**63**, o3851 [**doi:10.1107/S1600536807040482**]

5-Iodo-2-methyl-3-phenylsulfinyl-1-benzofuran

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

Comment

This work is related to an earlier communication on the synthesis and structure of a 5-iodo-1-benzofuran analogue, *viz*. 5-iodo-3-methylsulfinyl-2-phenyl-1-benzofuran (Choi *et al.*, 2007). Here we report the molecular and crystal structure of the title compound, 5-iodo-2-methyl-3-phenylsulfinyl-1-benzofuran (Fig. 1).

The benzofuran unit is essentially planar, with a mean deviation of 0.006 Å from the least-squares plane defined by the nine constituent atoms. The phenyl ring (C10–C15) is almost perpendicular to the plane of the benzofuran system [85.75 (7)°] and is tilted slightly towards it. The molecular packing (Fig. 2) is stabilized by a C—H···O hydrogen bond between the hydrogen of methyl group and the oxygen of the S═O unit (Table 1). The molecular packing (Fig. 2) is further stabilized by a halogen bond between the iodine atom and the oxygen of a neighbouring S═O unit, C—I···O2^{ii} (symmetry code as in Fig. 2). The observed I···O separation of 3.165 (2) Å and the nearly linear C—I···O angle of 165.55 (7)° are typical for such halogen bonds. A search of the Cambridge Structural Database (version 5.28; Allen, 2002) revealed 39 compounds with C—I···O═S contact distances equal to or less than 3.3 Å.

Experimental

3-Chloroperbenzoic acid (77%, 247 mg, 1.10 mmol) was added in small portions to a stirred solution of 5-iodo-2-methyl-3-phenylsulfanyl-1-benzofuran (366 mg, 1.0 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 (chloroform) to afford the title compound as a colorless solid [yield 81%, m.p. 441–442 K; R_f = 0.82 (hexane–ethyl acetate, 1:2 *v*/*v*)]. Single crystals suitable for X-ray diffraction were prepared 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) = 1.2Ueq(C) for aromatic H atoms and U_{iso}(H) = 1.5Ueq(C) for methyl H atoms. The deepest hole in the residual electron density is 0.70 Å from I.

Figures

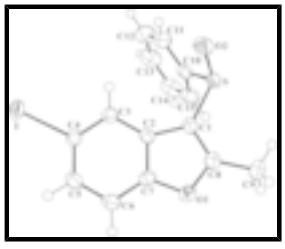


Fig. 1. The molecular structure of the title compound, showing displacement ellipsoides drawn at the 50% probability level.

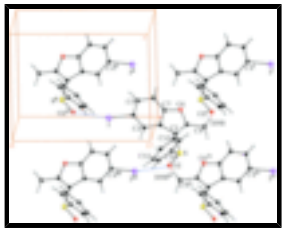


Fig. 2. The C—H···O hydrogen bond and I···O interaction (dotted lines) in the title compound. [Symmetry code: (i) *x*, −*y* − 1/2, *z* + 1/2; (ii) *x*, 1/2 − *y*, *z* + 1/2; (iii) *x*, 1/2 − *y*, *z* − 1/2; (iv) *x*, −*y* − 1/2, *z* − 1/2.]

5-Iodo-2-methyl-3-phenylsulfinyl-1-benzofuran

Crystal data

$C_{15}H_{11}I_1O_2S_1$	F_{000} = 744
M_r = 382.20	D_x = 1.810 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 6974 reflections
a = 12.9995 (6) Å	θ = 2.2–28.3º
b = 11.5063 (5) Å	μ = 2.43 mm^{-1}
c = 9.7556 (4) Å	*T* = 173 (2) K
β = 106.004 (1)º	Block, colourless
V = 1402.65 (11) Å^3	0.60 × 0.60 × 0.20 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3049 independent reflections
Radiation source: fine-focus sealed tube	2850 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.017
Detector resolution: 10.0 pixels mm^{-1}	θ_{max} = 27.0º
T = 173(2) K	θ_{min} = 2.4º
φ and ω scans	*h* = −14→16
Absorption correction: multi-scan (SADABS; Sheldrick, 1999)	*k* = −14→9
T_{min} = 0.248, T_{max} = 0.613	*l* = −12→12
8261 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.025$	H-atom parameters constrained
$wR(F^2) = 0.066$	$w = 1/[\sigma^2(F_o^2) + (0.0335P)^2 + 1.54P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
3049 reflections	$\Delta\rho_{max} = 0.92$ e Å^{-3}
173 parameters	$\Delta\rho_{min} = -1.04$ 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 $F^2 > 2$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}
I	0.778381 (14)	0.338404 (14)	0.039433 (19)	0.03671 (8)
S	0.70023 (5)	−0.16978 (5)	−0.27520 (6)	0.02966 (14)
O1	0.93626 (15)	−0.17067 (14)	0.07595 (18)	0.0302 (4)
O2	0.71346 (16)	−0.09253 (17)	−0.39233 (18)	0.0388 (4)
C1	0.79820 (19)	−0.1358 (2)	−0.1165 (2)	0.0263 (4)
C2	0.82329 (18)	−0.0273 (2)	−0.0382 (2)	0.0243 (4)
C3	0.78457 (18)	0.0870 (2)	−0.0546 (2)	0.0262 (4)
H3	0.7272	0.1094	−0.1338	0.031*
C4	0.8340 (2)	0.16585 (19)	0.0503 (3)	0.0279 (5)
C5	0.9196 (2)	0.1361 (2)	0.1672 (3)	0.0309 (5)
H5	0.9504	0.1933	0.2366	0.037*
C6	0.9596 (2)	0.0242 (2)	0.1824 (2)	0.0314 (5)
H6	1.0187	0.0027	0.2598	0.038*
C7	0.90908 (18)	−0.0551 (2)	0.0789 (2)	0.0262 (4)
C8	0.86830 (19)	−0.2171 (2)	−0.0443 (2)	0.0286 (5)
C9	0.8844 (2)	−0.3418 (2)	−0.0694 (3)	0.0359 (6)
H9A	0.8368	−0.3649	−0.1619	0.054*
H9B	0.8680	−0.3881	0.0063	0.054*

H9C	0.9589	−0.3549	−0.0690	0.054*
C10	0.58474 (19)	−0.1178 (2)	−0.2266 (2)	0.0292 (5)
C11	0.5264 (2)	−0.0268 (2)	−0.3007 (3)	0.0360 (5)
H11	0.5499	0.0125	−0.3723	0.043*
C12	0.4327 (2)	0.0070 (3)	−0.2695 (3)	0.0465 (7)
H12	0.3916	0.0696	−0.3203	0.056*
C13	0.3993 (2)	−0.0497 (3)	−0.1655 (3)	0.0503 (8)
H13	0.3352	−0.0263	−0.1447	0.060*
C14	0.4586 (3)	−0.1408 (3)	−0.0911 (3)	0.0489 (8)
H14	0.4354	−0.1791	−0.0187	0.059*
C15	0.5521 (2)	−0.1769 (3)	−0.1217 (3)	0.0396 (6)
H15	0.5926	−0.2403	−0.0721	0.047*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I	0.03934 (12)	0.02297 (10)	0.04727 (12)	−0.00219 (6)	0.01102 (8)	−0.00116 (6)
S	0.0380 (3)	0.0268 (3)	0.0238 (3)	−0.0043 (2)	0.0080 (2)	−0.0041 (2)
O1	0.0321 (9)	0.0279 (9)	0.0298 (8)	0.0025 (7)	0.0072 (7)	0.0032 (6)
O2	0.0492 (11)	0.0429 (11)	0.0277 (8)	−0.0024 (9)	0.0163 (8)	0.0026 (8)
C1	0.0299 (11)	0.0250 (11)	0.0253 (10)	−0.0027 (9)	0.0099 (9)	−0.0023 (9)
C2	0.0258 (10)	0.0258 (11)	0.0227 (9)	−0.0027 (9)	0.0090 (8)	−0.0007 (8)
C3	0.0261 (11)	0.0275 (11)	0.0250 (10)	−0.0010 (9)	0.0069 (8)	0.0012 (9)
C4	0.0318 (12)	0.0231 (11)	0.0302 (11)	−0.0041 (9)	0.0107 (10)	0.0005 (8)
C5	0.0345 (13)	0.0308 (12)	0.0259 (11)	−0.0089 (10)	0.0059 (9)	−0.0023 (9)
C6	0.0310 (12)	0.0353 (13)	0.0249 (10)	−0.0054 (10)	0.0025 (9)	0.0039 (9)
C7	0.0283 (11)	0.0257 (11)	0.0256 (10)	−0.0004 (9)	0.0089 (9)	0.0035 (9)
C8	0.0317 (12)	0.0284 (12)	0.0290 (11)	−0.0007 (9)	0.0138 (9)	−0.0002 (9)
C9	0.0434 (15)	0.0257 (13)	0.0431 (14)	0.0032 (10)	0.0191 (12)	0.0007 (10)
C10	0.0310 (12)	0.0309 (12)	0.0238 (10)	−0.0103 (10)	0.0043 (9)	−0.0044 (9)
C11	0.0342 (13)	0.0384 (14)	0.0336 (12)	−0.0061 (11)	0.0063 (10)	0.0036 (11)
C12	0.0328 (13)	0.0528 (18)	0.0498 (16)	0.0000 (12)	0.0046 (12)	0.0023 (14)
C13	0.0306 (13)	0.073 (2)	0.0468 (16)	−0.0076 (14)	0.0098 (12)	−0.0052 (15)
C14	0.0415 (16)	0.071 (2)	0.0369 (14)	−0.0201 (15)	0.0145 (12)	−0.0015 (14)
C15	0.0423 (15)	0.0454 (16)	0.0290 (12)	−0.0119 (12)	0.0066 (11)	0.0045 (11)

Geometric parameters (Å, °)

I—C4	2.106 (2)	C6—H6	0.9500
I—O2^{i}	3.165 (2)	C8—C9	1.479 (3)
S—O2	1.495 (2)	C9—H9A	0.9800
S—C1	1.756 (2)	C9—H9B	0.9800
S—C10	1.797 (3)	C9—H9C	0.9800
O1—C8	1.368 (3)	C10—C11	1.375 (4)
O1—C7	1.378 (3)	C10—C15	1.389 (3)
C1—C8	1.360 (3)	C11—C12	1.390 (4)
C1—C2	1.452 (3)	C11—H11	0.9500
C2—C7	1.396 (3)	C12—C13	1.374 (4)

C2—C3	1.402 (3)	C12—H12	0.9500
C3—C4	1.386 (3)	C13—C14	1.382 (5)
C3—H3	0.9500	C13—H13	0.9500
C4—C5	1.398 (4)	C14—C15	1.393 (5)
C5—C6	1.381 (4)	C14—H14	0.9500
C5—H5	0.9500	C15—H15	0.9500
C6—C7	1.385 (3)		
C4—I—O2^{i}	165.55 (7)	C1—C8—C9	133.4 (2)
O2—S—C1	109.5 (1)	O1—C8—C9	115.7 (2)
O2—S—C10	106.5 (1)	C8—C9—H9A	109.5
C1—S—C10	98.6 (1)	C8—C9—H9B	109.5
C8—O1—C7	106.6 (2)	H9A—C9—H9B	109.5
C8—C1—C2	107.3 (2)	C8—C9—H9C	109.5
C8—C1—S	121.5 (2)	H9A—C9—H9C	109.5
C2—C1—S	131.2 (2)	H9B—C9—H9C	109.5
C7—C2—C3	119.3 (2)	C11—C10—C15	121.6 (3)
C7—C2—C1	104.3 (2)	C11—C10—S	119.6 (2)
C3—C2—C1	136.5 (2)	C15—C10—S	118.6 (2)
C4—C3—C2	116.8 (2)	C10—C11—C12	119.2 (3)
C4—C3—H3	121.6	C10—C11—H11	120.4
C2—C3—H3	121.6	C12—C11—H11	120.4
C3—C4—C5	123.0 (2)	C13—C12—C11	120.2 (3)
C3—C4—I	119.8 (2)	C13—C12—H12	119.9
C5—C4—I	117.20 (17)	C11—C12—H12	119.9
C6—C5—C4	120.5 (2)	C12—C13—C14	120.3 (3)
C6—C5—H5	119.8	C12—C13—H13	119.9
C4—C5—H5	119.8	C14—C13—H13	119.9
C5—C6—C7	116.6 (2)	C13—C14—C15	120.4 (3)
C5—C6—H6	121.7	C13—C14—H14	119.8
C7—C6—H6	121.7	C15—C14—H14	119.8
O1—C7—C6	125.3 (2)	C10—C15—C14	118.3 (3)
O1—C7—C2	110.8 (2)	C10—C15—H15	120.9
C6—C7—C2	123.8 (2)	C14—C15—H15	120.9
C1—C8—O1	110.9 (2)		
O2—S—C1—C8	−122.3 (2)	C1—C2—C7—O1	−0.1 (2)
C10—S—C1—C8	126.7 (2)	C3—C2—C7—C6	0.1 (3)
O2—S—C1—C2	57.2 (2)	C1—C2—C7—C6	179.4 (2)
C10—S—C1—C2	−53.8 (2)	C2—C1—C8—O1	0.8 (3)
C8—C1—C2—C7	−0.4 (2)	S—C1—C8—O1	−179.60 (16)
S—C1—C2—C7	−179.97 (18)	C2—C1—C8—C9	−179.6 (2)
C8—C1—C2—C3	178.8 (2)	S—C1—C8—C9	0.0 (4)
S—C1—C2—C3	−0.8 (4)	C7—O1—C8—C1	−0.9 (2)
C7—C2—C3—C4	−1.1 (3)	C7—O1—C8—C9	179.4 (2)
C1—C2—C3—C4	179.8 (2)	O2—S—C10—C11	3.0 (2)
C2—C3—C4—C5	0.9 (3)	C1—S—C10—C11	116.3 (2)
C2—C3—C4—I	−177.91 (15)	O2—S—C10—C15	178.09 (19)
O2^{i}—I—C4—C3	147.5 (2)	C1—S—C10—C15	−68.6 (2)
O2^{i}—I—C4—C5	−31.4 (4)	C15—C10—C11—C12	0.2 (4)

C3—C4—C5—C6	0.4 (4)	S—C10—C11—C12	175.1 (2)
I—C4—C5—C6	179.26 (18)	C10—C11—C12—C13	0.2 (4)
C4—C5—C6—C7	−1.4 (3)	C11—C12—C13—C14	0.0 (5)
C8—O1—C7—C6	−178.9 (2)	C12—C13—C14—C15	−0.7 (5)
C8—O1—C7—C2	0.6 (2)	C11—C10—C15—C14	−0.8 (4)
C5—C6—C7—O1	−179.3 (2)	S—C10—C15—C14	−175.9 (2)
C5—C6—C7—C2	1.2 (3)	C13—C14—C15—C10	1.1 (4)
C3—C2—C7—O1	−179.44 (19)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9B···O2^{ii}	0.98	2.48	3.257 (3)	136

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

Fig. 1

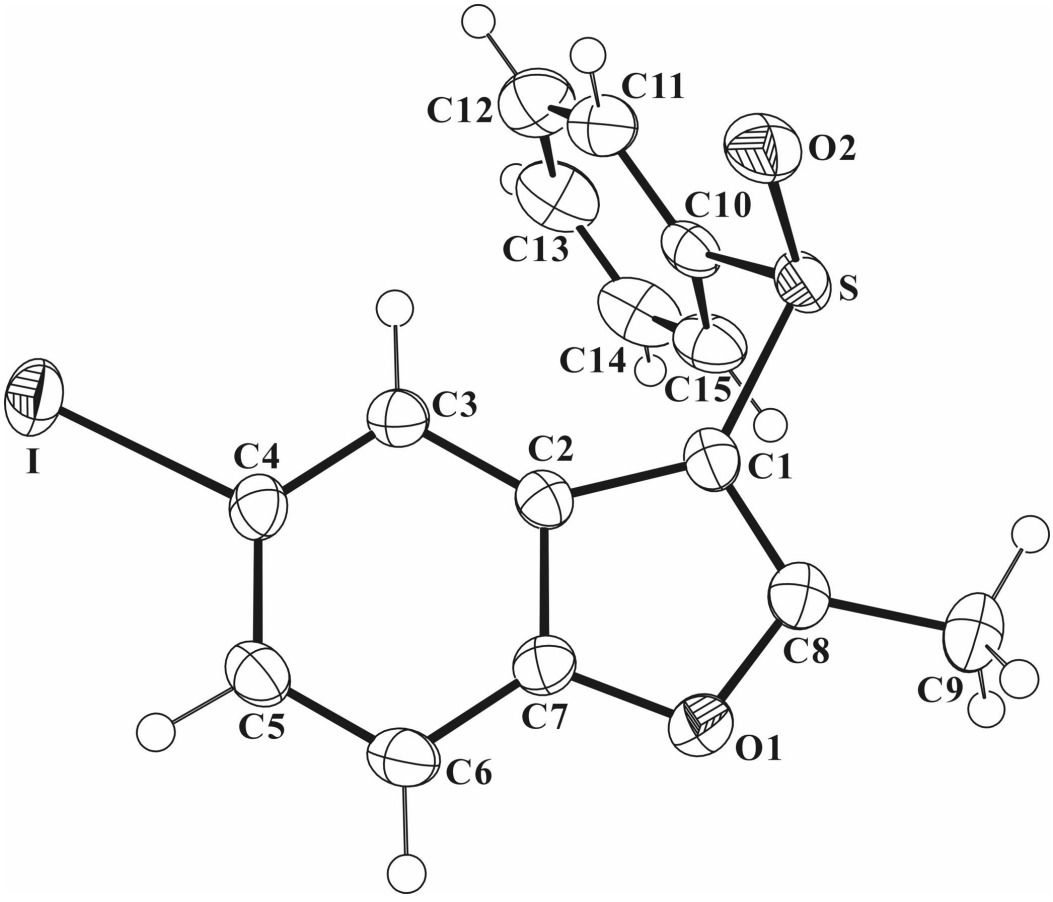
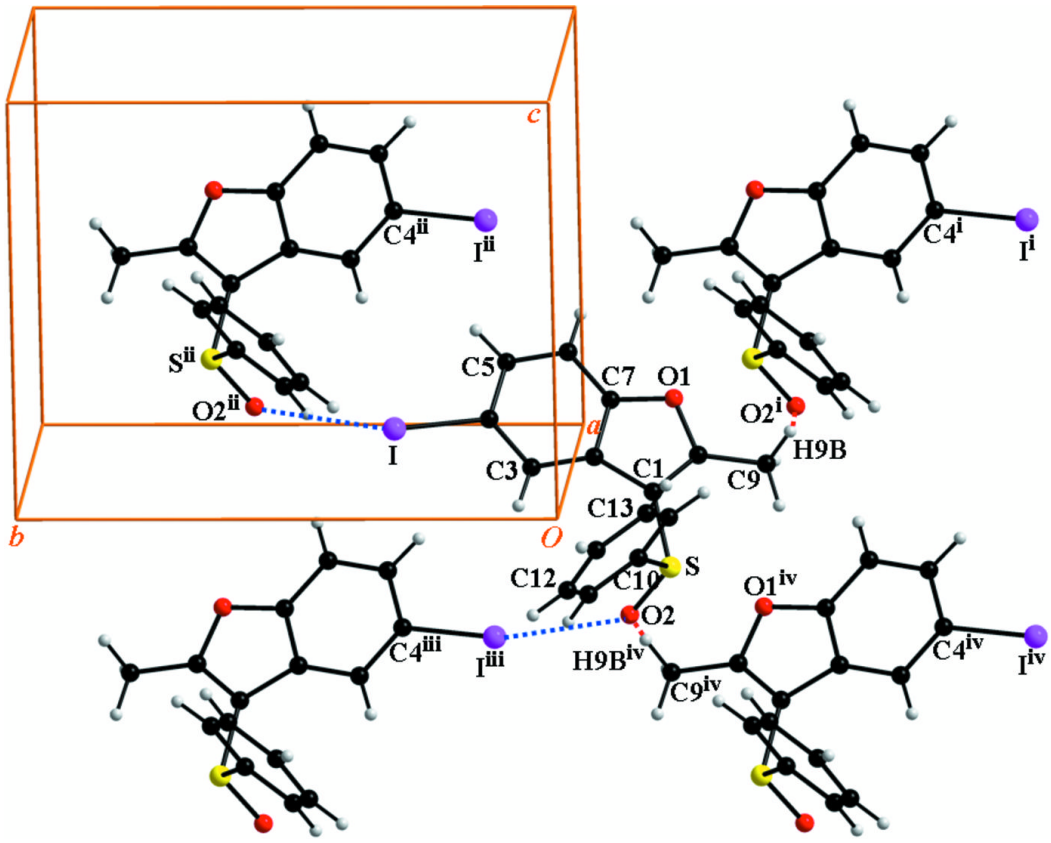


Fig. 2


c
b
O
a
C4ii
Iii
Sii
O2ii
I
C5
C7
O1
C3
C1
C13
C12
C10
S
O2
H9Biv
C9iv
O1iv
C4iv
Iiv
C4iii
Iiii
C4i
Ii
O2i
H9B
C9