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3-Ethylsulfinyl-5-fluoro-2-(4-iodophenyl)-1-benzofuran

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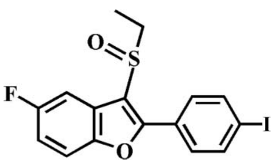
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.024; wR factor = 0.062; data-to-parameter ratio = 17.1.

In the title compound, $C_{16}H_{12}FIO_2S$, the 4-iodophenyl ring is rotated slightly out of the benzofuran plane, as indicated by the dihedral angle of 11.41 (7)°. The crystal structure is stabilized by an intermolecular π–π interaction between the benzene and 4-iodophenyl rings [centroid–centroid distance = 3.757 (3) Å]. The crystal structure also exhibits a weak intermolecular C—H···O hydrogen bond and a short I···O [3.2575 (16) Å] contact.

Related literature

For the pharmacological activity of benzofuran compounds, see: Aslam *et al.* (2006); Galal *et al.* (2009); Khan *et al.* (2005). For natural products with benzofuran rings, see: Akgul & Anil (2003); Soekamto *et al.* (2003). For structures of related 3-ethylsulfinyl-2-(4-fluorophenyl)-5-halo-1-benzofuran derivatives, see: Choi *et al.* (2010*a*,*b*,*c*). For a review of halogen bonding, see: Politzer *et al.* (2007).



Experimental

Crystal data

$C_{16}H_{12}FIO_2S$	M_r = 414.22
Monoclinic, $P2_1/n$	Z = 4
a = 7.5384 (4) Å	Mo $K\alpha$ radiation
b = 9.4104 (5) Å	μ = 2.39 mm^{-1}
c = 20.3049 (9) Å	T = 173 K
β = 96.298 (2)°	0.24 × 0.18 × 0.16 mm
V = 1431.72 (12) Å^3	

Data collection

Bruker SMART APEXII CCD diffractometer	12915 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)	3265 independent reflections
T_{min} = 0.658, T_{max} = 0.680	3061 reflections with $I > 2\sigma(I)$
	R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024	191 parameters
$wR(F^2)$ = 0.062	H-atom parameters constrained
S = 1.13	$\Delta\rho_{max}$ = 0.53 e Å^{-3}
3265 reflections	$\Delta\rho_{min}$ = −0.78 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}	0.97	2.59	3.337 (3)	134

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); 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: IS2566).

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supplementary materials

Acta Cryst. (2010). E**66**, o1862 [**doi:10.1107/S1600536810024736**]

3-Ethylsulfinyl-5-fluoro-2-(4-iodophenyl)-1-benzofuran

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Comment

Compounds involving a benzofuran moiety show potent pharmacological properties such as antifungal (Aslam *et al.*, 2006), antitumor and antiviral (Galal *et al.*, 2009), antimicrobial (Khan *et al.*, 2005) activities. These compounds occur widely in nature (Akgul & Anil, 2003; Soekamto *et al.*, 2003). As a part of our ongoing studies of the substituent effect on the solid state structures of 3-ethylsulfinyl-2-(4-fluorophenyl)-5-halo-1-benzofuran analogues (Choi *et al.*, 2010*a,b,c*), we report the crystal structure of the title compound (Fig. 1).

The benzofuran unit is essentially planar, with a mean deviation of 0.011 (2)Å from the least-squares plane defined by the nine constituent atoms. The dihedral angle formed by the benzofuran plane and the 4-iodophenyl ring is 11.41 (7)°. The crystal packing (Fig. 2) is stabilized by aromatic π–π interactions between the benzene and the 4-iodophenyl rings of the neighbouring molecules, with a $Cg1\cdots Cg2^{iii}$ distance of 3.757 (3) Å ($Cg1$ and $Cg2$ are the centroids of the C2–C7 benzene ring and the C11–C16 4-iodophenyl ring, respectively). The molecular packing (Fig. 2) is further stabilized by a weak intermolecular C—H···O hydrogen bond between the methylene H atom of the ethyl group and the oxygen of the S=O unit, with a C9—H9B···O2^{i} (Table 1). The crystal packing (Fig. 2) also exhibits an I···O halogen bonding between the iodine and the oxygen of the S=O unit [I···O2^{ii} = 3.2575 (16) Å; C14—I···O2^{ii} = 153.53 (6)°] (Politzer *et al.*, 2007).

Experimental

77% 3-Chloroperoxybenzoic acid (202 mg, 0.9 mmol) was added in small portions to a stirred solution of 3-ethylsulfanyl-5-fluoro-2-(4-iodophenyl)-1-benzofuran (318 mg, 0.8 mmol) in dichloromethane (40 mL) at 273 K. After being stirred at room temperature for 4h, the mixture was washed with saturated sodium bicarbonate solution and the organic layer was separated, dried over magnesium sulfate, filtered and concentrated at reduced pressure. The residue was purified by column chromatography (hexane–ethyl acetate, 1:1 *v*/*v*) to afford the title compound as a colorless solid [yield 78%, m.p. 434–435 K; R_f = 0.59 (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 tetrahydrofuran at room temperature.

Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å for aryl, 0.97 Å for methylene, and 0.96 Å for methyl H atoms, and with U_{iso}(H) = 1.2U_{eq}(C) for aryl and methylene H atoms, and 1.5U_{eq}(C) for methyl H atoms.

Figures

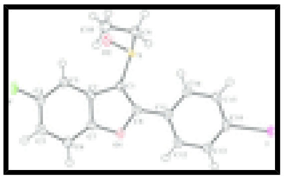


Fig. 1. The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as a small spheres of arbitrary radius.

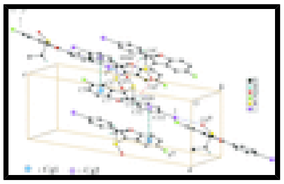


Fig. 2. π–π, C—H···O, and I···O interactions (dotted lines) in the crystal structure of the title compound. Cg1 and Cg2 denote the ring centroids. [Symmetry codes: (i) $-x, -y+2, -z+1$; (ii) $x+1/2, -y+1/2, z-1/2$; (iii) $-x, -y+1, -z+1$; (iv) $-x+1, -y+1, -z+1$; (v) $x-1/2, -y+3/2, z+1/2$.]

3-Ethylsulfinyl-5-fluoro-2-(4-iodophenyl)-1-benzofuran

Crystal data

$C_{16}H_{12}FIO_2S$

$M_r = 414.22$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 7.5384$ (4) Å

$b = 9.4104$ (5) Å

$c = 20.3049$ (9) Å

$\beta = 96.298$ (2)°

$V = 1431.72$ (12) Å^3

$Z = 4$

$F(000) = 808$

$D_x = 1.922$ Mg m^{-3}

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 8330 reflections

$\theta = 2.4$–27.5°

$\mu = 2.39$ mm^{-1}

$T = 173$ K

Block, colourless

0.24 × 0.18 × 0.16 mm

Data collection

Bruker SMART APEXII CCD diffractometer

Radiation source: rotating anode

graphite multilayer

Detector resolution: 10.0 pixels mm^{-1}

φ and ω scans

Absorption correction: multi-scan (*SADABS*; Bruker, 2009)

$T_{min} = 0.658$, $T_{max} = 0.680$

12915 measured reflections

3265 independent reflections

3061 reflections with $I > 2\sigma(I)$

$R_{int} = 0.030$

$\theta_{max} = 27.5°$, $\theta_{min} = 2.0°$

$h = -9 \rightarrow 9$

$k = -11 \rightarrow 12$

$l = -26 \rightarrow 26$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.024$

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

$wR(F^2) = 0.062$	H-atom parameters constrained
$S = 1.13$	$w = 1/[\sigma^2(F_o^2) + (0.0234P)^2 + 1.1834P]$ where $P = (F_o^2 + 2F_c^2)/3$
3265 reflections	$(\Delta/\sigma)_{max} = 0.001$
191 parameters	$\Delta\rho_{max} = 0.53$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.78$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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}
I	0.410489 (19)	0.499975 (13)	0.189463 (7)	0.02345 (7)
S	0.10698 (7)	0.80859 (5)	0.50166 (2)	0.01980 (11)
F	0.07034 (19)	0.56507 (16)	0.76127 (6)	0.0317 (3)
O1	0.27247 (19)	0.40664 (15)	0.52431 (7)	0.0203 (3)
O2	−0.0186 (2)	0.85501 (17)	0.54905 (8)	0.0283 (3)
C1	0.1851 (3)	0.6367 (2)	0.52771 (10)	0.0183 (4)
C2	0.1707 (3)	0.5743 (2)	0.59203 (10)	0.0184 (4)
C3	0.1180 (3)	0.6214 (2)	0.65229 (10)	0.0210 (4)
H3	0.0824	0.7147	0.6583	0.025*
C4	0.1218 (3)	0.5224 (2)	0.70199 (11)	0.0227 (4)
C5	0.1728 (3)	0.3806 (2)	0.69609 (11)	0.0246 (4)
H5	0.1710	0.3185	0.7316	0.030*
C6	0.2257 (3)	0.3339 (2)	0.63685 (11)	0.0234 (4)
H6	0.2605	0.2403	0.6311	0.028*
C7	0.2246 (3)	0.4326 (2)	0.58651 (10)	0.0191 (4)
C8	0.2457 (3)	0.5318 (2)	0.48905 (10)	0.0181 (4)
C9	0.3111 (3)	0.9094 (2)	0.52147 (11)	0.0242 (4)
H9A	0.4062	0.8643	0.5006	0.029*
H9B	0.2944	1.0041	0.5030	0.029*
C10	0.3674 (3)	0.9214 (3)	0.59512 (11)	0.0296 (5)
H10A	0.2726	0.9633	0.6164	0.044*
H10B	0.4720	0.9799	0.6025	0.044*
H10C	0.3934	0.8285	0.6132	0.044*
C11	0.2883 (3)	0.5241 (2)	0.42083 (11)	0.0185 (4)
C12	0.3152 (3)	0.3921 (2)	0.39142 (10)	0.0215 (4)

H12	0.3078	0.3092	0.4159	0.026*
C13	0.3528 (3)	0.3837 (2)	0.32607 (11)	0.0229 (4)
H13	0.3720	0.2957	0.3072	0.028*
C14	0.3616 (3)	0.5066 (2)	0.28903 (11)	0.0199 (4)
C15	0.3361 (3)	0.6385 (2)	0.31721 (11)	0.0233 (4)
H15	0.3432	0.7209	0.2923	0.028*
C16	0.3001 (3)	0.6469 (2)	0.38255 (11)	0.0234 (4)
H16	0.2834	0.7354	0.4013	0.028*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I	0.02965 (10)	0.02541 (10)	0.01641 (9)	−0.00231 (5)	0.00756 (6)	−0.00183 (4)
S	0.0218 (2)	0.0183 (2)	0.0192 (2)	0.00129 (18)	0.00201 (18)	0.00248 (18)
F	0.0415 (8)	0.0402 (8)	0.0148 (6)	0.0028 (6)	0.0099 (5)	0.0013 (5)
O1	0.0263 (7)	0.0193 (7)	0.0158 (7)	0.0016 (6)	0.0041 (6)	0.0012 (5)
O2	0.0297 (8)	0.0282 (8)	0.0282 (8)	0.0049 (7)	0.0091 (7)	0.0018 (7)
C1	0.0197 (9)	0.0178 (9)	0.0173 (9)	0.0000 (7)	0.0016 (7)	0.0018 (7)
C2	0.0177 (9)	0.0188 (10)	0.0184 (9)	−0.0002 (7)	0.0012 (7)	0.0009 (8)
C3	0.0220 (10)	0.0239 (10)	0.0173 (9)	0.0010 (8)	0.0034 (8)	−0.0009 (8)
C4	0.0232 (10)	0.0305 (11)	0.0149 (10)	−0.0011 (8)	0.0050 (8)	−0.0007 (8)
C5	0.0273 (10)	0.0264 (11)	0.0200 (10)	−0.0012 (9)	0.0022 (8)	0.0078 (8)
C6	0.0264 (10)	0.0206 (10)	0.0230 (10)	0.0005 (8)	0.0018 (8)	0.0033 (8)
C7	0.0196 (9)	0.0204 (10)	0.0175 (9)	−0.0008 (8)	0.0026 (8)	−0.0007 (8)
C8	0.0177 (9)	0.0187 (9)	0.0177 (10)	−0.0010 (7)	0.0010 (8)	0.0023 (8)
C9	0.0254 (10)	0.0225 (10)	0.0252 (11)	−0.0049 (8)	0.0048 (9)	0.0014 (8)
C10	0.0294 (11)	0.0307 (12)	0.0283 (12)	−0.0051 (9)	0.0015 (9)	−0.0041 (9)
C11	0.0165 (9)	0.0230 (10)	0.0163 (10)	0.0000 (7)	0.0026 (7)	−0.0002 (8)
C12	0.0264 (10)	0.0209 (10)	0.0179 (10)	0.0010 (8)	0.0055 (8)	0.0017 (8)
C13	0.0278 (10)	0.0201 (10)	0.0211 (10)	0.0022 (8)	0.0042 (8)	−0.0021 (8)
C14	0.0206 (10)	0.0249 (11)	0.0144 (10)	−0.0008 (7)	0.0026 (8)	−0.0017 (7)
C15	0.0314 (11)	0.0201 (10)	0.0189 (10)	−0.0003 (8)	0.0050 (8)	0.0030 (8)
C16	0.0321 (11)	0.0199 (10)	0.0192 (10)	0.0009 (8)	0.0067 (8)	−0.0023 (8)

Geometric parameters (Å, °)

I—C14	2.095 (2)	C6—H6	0.9300
I—O2^{i}	3.2575 (16)	C8—C11	1.458 (3)
S—O2	1.4876 (17)	C9—C10	1.513 (3)
S—C1	1.782 (2)	C9—H9A	0.9700
S—C9	1.815 (2)	C9—H9B	0.9700
F—C4	1.365 (3)	C10—H10A	0.9600
O1—C7	1.373 (2)	C10—H10B	0.9600
O1—C8	1.381 (2)	C10—H10C	0.9600
C1—C8	1.370 (3)	C11—C16	1.401 (3)
C1—C2	1.447 (3)	C11—C12	1.403 (3)
C2—C3	1.399 (3)	C12—C13	1.389 (3)
C2—C7	1.402 (3)	C12—H12	0.9300

C3—C4	1.372 (3)	C13—C14	1.385 (3)
C3—H3	0.9300	C13—H13	0.9300
C4—C5	1.397 (3)	C14—C15	1.389 (3)
C5—C6	1.380 (3)	C15—C16	1.385 (3)
C5—H5	0.9300	C15—H15	0.9300
C6—C7	1.380 (3)	C16—H16	0.9300

C14—I—O2^{i}	153.53 (6)	C10—C9—H9A	108.9
O2—S—C1	106.72 (9)	S—C9—H9A	108.9
O2—S—C9	106.71 (10)	C10—C9—H9B	108.9
C1—S—C9	99.45 (10)	S—C9—H9B	108.9
C7—O1—C8	106.66 (15)	H9A—C9—H9B	107.7
C8—C1—C2	106.71 (18)	C9—C10—H10A	109.5
C8—C1—S	127.16 (16)	C9—C10—H10B	109.5
C2—C1—S	125.55 (15)	H10A—C10—H10B	109.5
C3—C2—C7	118.95 (18)	C9—C10—H10C	109.5
C3—C2—C1	135.82 (19)	H10A—C10—H10C	109.5
C7—C2—C1	105.23 (17)	H10B—C10—H10C	109.5
C4—C3—C2	116.3 (2)	C16—C11—C12	118.3 (2)
C4—C3—H3	121.9	C16—C11—C8	121.35 (19)
C2—C3—H3	121.9	C12—C11—C8	120.38 (19)
F—C4—C3	117.6 (2)	C13—C12—C11	120.73 (19)
F—C4—C5	117.6 (2)	C13—C12—H12	119.6
C3—C4—C5	124.7 (2)	C11—C12—H12	119.6
C6—C5—C4	119.2 (2)	C14—C13—C12	119.9 (2)
C6—C5—H5	120.4	C14—C13—H13	120.1
C4—C5—H5	120.4	C12—C13—H13	120.1
C5—C6—C7	116.9 (2)	C13—C14—C15	120.4 (2)
C5—C6—H6	121.5	C13—C14—I	121.53 (15)
C7—C6—H6	121.5	C15—C14—I	118.10 (15)
O1—C7—C6	125.48 (19)	C16—C15—C14	119.7 (2)
O1—C7—C2	110.53 (17)	C16—C15—H15	120.1
C6—C7—C2	123.98 (19)	C14—C15—H15	120.1
C1—C8—O1	110.86 (18)	C15—C16—C11	121.0 (2)
C1—C8—C11	134.61 (19)	C15—C16—H16	119.5
O1—C8—C11	114.53 (18)	C11—C16—H16	119.5
C10—C9—S	113.40 (16)		

O2—S—C1—C8	153.69 (18)	C2—C1—C8—O1	−0.4 (2)
C9—S—C1—C8	−95.6 (2)	S—C1—C8—O1	−171.94 (14)
O2—S—C1—C2	−16.3 (2)	C2—C1—C8—C11	−179.9 (2)
C9—S—C1—C2	94.44 (18)	S—C1—C8—C11	8.6 (4)
C8—C1—C2—C3	−179.7 (2)	C7—O1—C8—C1	1.0 (2)
S—C1—C2—C3	−8.0 (3)	C7—O1—C8—C11	−179.43 (17)
C8—C1—C2—C7	−0.3 (2)	O2—S—C9—C10	42.74 (19)
S—C1—C2—C7	171.38 (15)	C1—S—C9—C10	−68.02 (18)
C7—C2—C3—C4	−0.6 (3)	C1—C8—C11—C16	11.3 (4)
C1—C2—C3—C4	178.7 (2)	O1—C8—C11—C16	−168.10 (19)
C2—C3—C4—F	−179.70 (18)	C1—C8—C11—C12	−167.4 (2)
C2—C3—C4—C5	−0.4 (3)	O1—C8—C11—C12	13.2 (3)

F—C4—C5—C6	−179.96 (19)	C16—C11—C12—C13	0.1 (3)
C3—C4—C5—C6	0.8 (4)	C8—C11—C12—C13	178.88 (19)
C4—C5—C6—C7	0.0 (3)	C11—C12—C13—C14	−0.9 (3)
C8—O1—C7—C6	177.8 (2)	C12—C13—C14—C15	1.1 (3)
C8—O1—C7—C2	−1.2 (2)	C12—C13—C14—I	−178.22 (16)
C5—C6—C7—O1	−179.99 (19)	C13—C14—C15—C16	−0.6 (4)
C5—C6—C7—C2	−1.1 (3)	I—C14—C15—C16	178.77 (17)
C3—C2—C7—O1	−179.52 (17)	C14—C15—C16—C11	−0.2 (3)
C1—C2—C7—O1	1.0 (2)	C12—C11—C16—C15	0.4 (3)
C3—C2—C7—C6	1.4 (3)	C8—C11—C16—C15	−178.3 (2)
C1—C2—C7—C6	−178.09 (19)		

Symmetry codes: (i) *x*+1/2, −*y*+3/2, *z*−1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9B···O2^{ii}	0.97	2.59	3.337 (3)	134

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

Fig. 1

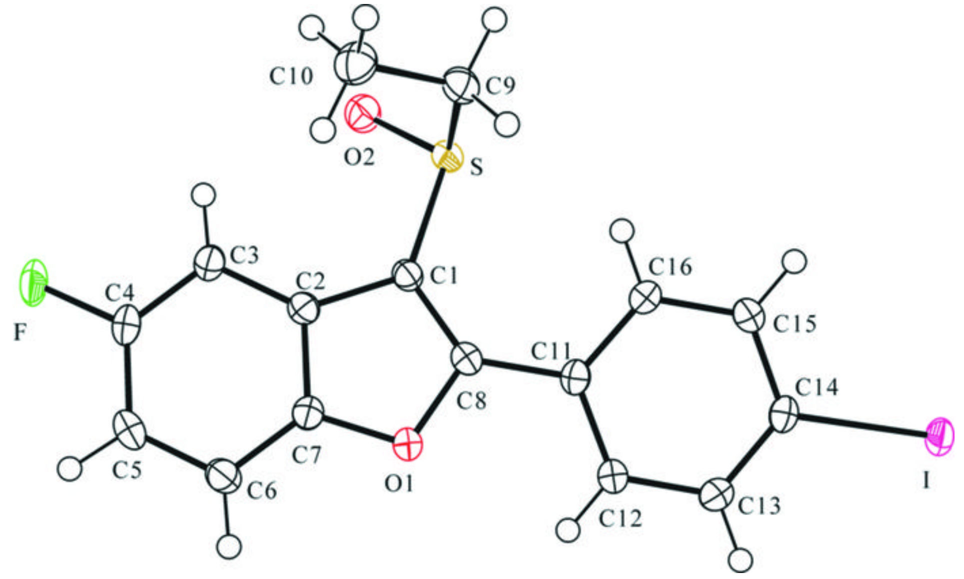
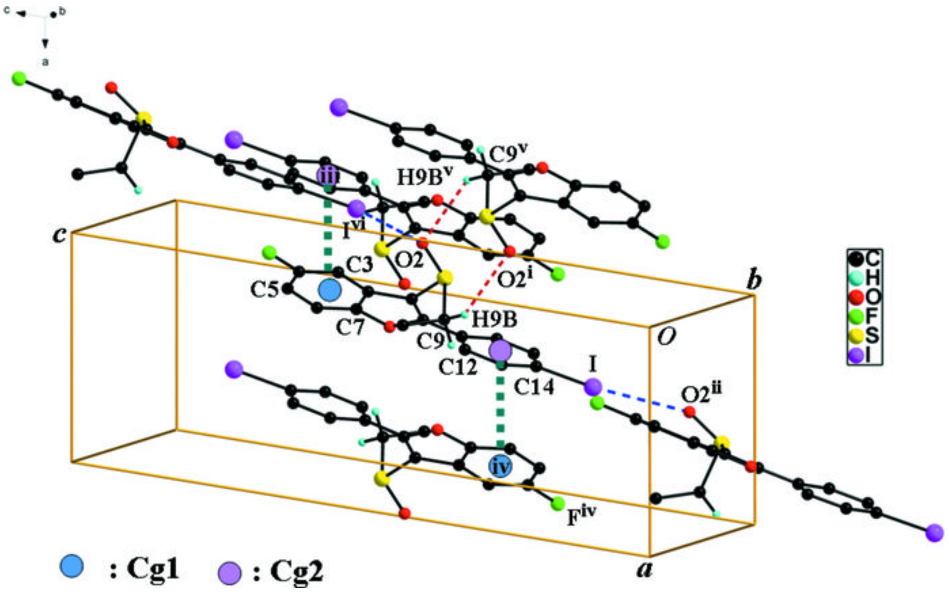


Fig. 2


c
b
a
C9v
H9Bv
iii
Ivi
C3
O2
O2i
C5
C7
C9
H9B
C12
C14
I
O2ii
iv
Fiv
c
b
O
a
C
H
O
F
S
I
: Cg1
: Cg2