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

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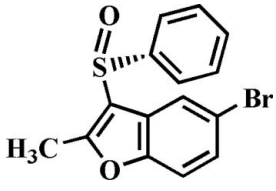
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.030; wR factor = 0.085; data-to-parameter ratio = 17.1.

The title compound, $C_{15}H_{11}BrO_2S$, was prepared by the oxidation of 5-bromo-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 fragment. The phenyl ring is almost perpendicular to the plane of the benzofuran fragment [84.61 (6)°] and is tilted slightly towards it. The crystal structure is stabilized by intermolecular aromatic π–π interactions, with a centroid–centroid distance of 3.622 (3) Å for the furan and benzene rings, C—H···O hydrogen bonds and a Br···O (π-antibondng of S=O to π-non-bonding of Br) interaction, with a distance of 3.204 (2) Å.

Related literature

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



Experimental

Crystal data

$C_{15}H_{11}BrO_2S$
M_r = 335.21
Monoclinic, $P2_1/c$
a = 12.8097 (7) Å
b = 11.1838 (6) Å
c = 9.9046 (5) Å
β = 106.084 (1)°
V = 1363.40 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.16 mm^{-1}
T = 173 (2) K
0.42 × 0.40 × 0.12 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1999)
T_{min} = 0.275, T_{max} = 0.692
8012 measured reflections
2964 independent reflections
2534 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.085
S = 1.05
2964 reflections
173 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.89 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O1^{i}	0.95	2.49	3.420 (3)	166

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

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WK2060).

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  doi:10.1107/S1600536807028504

supplementary materials

Acta Cryst. (2007). E**63**, o3204 [**doi:10.1107/S1600536807028504**]

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

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Comment

As part of our continuing studies on the synthesis and structure of 5-bromo-1-benzofuran analogues, the crystal structures of 5-bromo-2-methyl-3-methylsulfinyl-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 0.008 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 (84.61 (6) degrees and is tilted slightly towards it. The molecular packing (Fig. 2) is stabilized by π–π stacking interactions between adjacent benzofuran units. The *Cg*1···*Cg*2 distance is 3.622 (3) Å (*Cg*1 and *Cg*2 are of the centroids of the O1/C8/C1/C2/C7 and C2—C7 rings; symmetry code as in Fig. 2). Further stability comes from weak C—H···O hydrogen bond in Table 1, and Br···O2^{i} interaction at 3.204 (2) Å {Symmetry code; (i): *x*, 1/2 − *y*, 1/2 − *z*.}

Experimental

3-Chloroperbenzoic acid (77%, 291 mg, 1.30 mmol) was added in small portions to a stirred solution of 5-bromo-2-methyl-3-phenylsulfanyl-1-benzofuran (383 mg, 1.20 mmol) in dichloromethane (30 ml) at 273 K. After being stirred at room temperature for 2hrs, 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 (hexane-ethyl acetate, 1:1 *v*/*v*) to afford the title compound as a colorless solid [yield 89%, m.p. 413–414 K; R_f = 0.56 (hexane-ethyl acetate, 1:1 *v*/*v*)]. Single crystals suitable for X-ray diffraction were prepared by slow evaporation of a dilute solution of title compound (I) in benzene 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) = 1.2Ueq(C) for aromatic H atoms, and U_{iso}(H) = 1.5Ueq(C) for methyl H atoms. The highest peak in the difference map is 0.74 Å from Br and the largest hole is 0.75Å from Br.

Figures

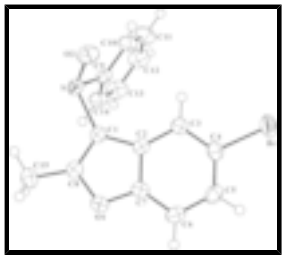


Fig. 1. The molecular structure of (I), showing displacement ellipsoides drawn at the 50% probability level.

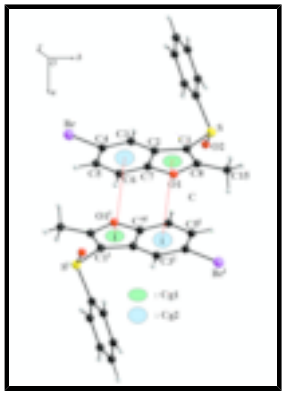


Fig. 2. π–π stacking interactions (dotted lines) in (I). *Cg* denotes the ring centroid. [Symmetry code: (i) 1 − *x*, 1 − *y*, −*z*.]

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

Crystal data

$C_{15}H_{11}BrO_2S$	F_{000} = 672
M_r = 335.21	D_x = 1.633 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -p_2ybc	Cell parameters from 4433 reflections
a = 12.8097 (7) Å	θ = 2.5–28.1º
b = 11.1838 (6) Å	μ = 3.16 mm^{-1}
c = 9.9046 (5) Å	*T* = 173 (2) K
β = 106.084 (1)º	Block, colorless
V = 1363.40 (13) Å^3	0.42 × 0.40 × 0.12 mm
Z = 4	

Data collection

Bruker SMART CCD diffractometer	2964 independent reflections
Radiation source: fine-focus sealed tube	2534 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.020
Detector resolution: 10.00 pixels mm^{-1}	θ_{max} = 27.0º
T = 173(2) K	θ_{min} = 2.5º
φ and ω scans	*h* = −12→16
Absorption correction: multi-scan	*k* = −14→13

(SADABS; Sheldrick, 1999)
T_{min} = 0.275, T_{max} = 0.692
8012 measured reflections

l = −12→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.085
S = 1.05
2964 reflections
173 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
$w = 1/[\sigma^2(F_o^2) + (0.0451P)^2 + 0.7554P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.89 e Å^{-3}
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}
Br	0.28113 (2)	0.16625 (2)	0.03045 (3)	0.04675 (11)
S	0.20038 (4)	0.67420 (5)	−0.27287 (5)	0.02839 (13)
O1	0.43606 (12)	0.67227 (13)	0.07797 (15)	0.0301 (3)
O2	0.21425 (14)	0.59407 (16)	−0.38714 (16)	0.0400 (4)
C1	0.29805 (16)	0.63812 (19)	−0.1152 (2)	0.0257 (4)
C2	0.32238 (15)	0.52620 (18)	−0.0398 (2)	0.0245 (4)
C3	0.28368 (16)	0.40820 (18)	−0.0591 (2)	0.0275 (4)
H3	0.2270	0.3854	−0.1393	0.033*
C4	0.33223 (19)	0.32668 (18)	0.0444 (2)	0.0315 (5)
C5	0.41681 (19)	0.3558 (2)	0.1623 (2)	0.0337 (5)
H5	0.4466	0.2963	0.2305	0.040*
C6	0.45763 (18)	0.4713 (2)	0.1802 (2)	0.0317 (5)
H6	0.5165	0.4929	0.2583	0.038*
C7	0.40803 (16)	0.55317 (19)	0.0783 (2)	0.0268 (4)
C8	0.36849 (17)	0.72134 (19)	−0.0416 (2)	0.0275 (4)

C9	0.08227 (16)	0.62234 (19)	−0.2262 (2)	0.0270 (4)
C10	0.02089 (18)	0.5314 (2)	−0.3034 (2)	0.0351 (5)
H10	0.0439	0.4924	−0.3754	0.042*
C11	−0.0750 (2)	0.4979 (3)	−0.2743 (3)	0.0456 (6)
H11	−0.1182	0.4357	−0.3270	0.055*
C12	−0.10762 (19)	0.5542 (3)	−0.1696 (3)	0.0462 (6)
H12	−0.1734	0.5308	−0.1504	0.055*
C13	−0.0453 (2)	0.6448 (3)	−0.0920 (3)	0.0443 (6)
H13	−0.0680	0.6826	−0.0191	0.053*
C14	0.05024 (19)	0.6807 (2)	−0.1201 (2)	0.0359 (5)
H14	0.0928	0.7437	−0.0682	0.043*
C15	0.3859 (2)	0.84977 (19)	−0.0639 (3)	0.0354 (5)
H15A	0.3455	0.8723	−0.1597	0.053*
H15B	0.4636	0.8646	−0.0503	0.053*
H15C	0.3603	0.8974	0.0036	0.053*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.04578 (17)	0.02535 (14)	0.0674 (2)	0.00332 (9)	0.01274 (13)	0.00219 (10)
S	0.0311 (3)	0.0303 (3)	0.0241 (2)	0.0034 (2)	0.0083 (2)	0.00379 (19)
O1	0.0279 (8)	0.0317 (8)	0.0296 (7)	−0.0025 (6)	0.0062 (6)	−0.0040 (6)
O2	0.0448 (9)	0.0505 (10)	0.0284 (8)	0.0034 (8)	0.0161 (7)	−0.0036 (7)
C1	0.0240 (9)	0.0288 (10)	0.0257 (10)	0.0017 (8)	0.0091 (8)	0.0021 (8)
C2	0.0217 (9)	0.0289 (10)	0.0243 (9)	0.0035 (8)	0.0086 (7)	0.0001 (8)
C3	0.0244 (10)	0.0287 (10)	0.0297 (10)	0.0015 (8)	0.0078 (8)	−0.0021 (8)
C4	0.0317 (11)	0.0258 (10)	0.0391 (12)	0.0048 (8)	0.0131 (9)	−0.0014 (8)
C5	0.0357 (12)	0.0335 (11)	0.0315 (11)	0.0122 (9)	0.0086 (9)	0.0041 (9)
C6	0.0299 (11)	0.0385 (12)	0.0251 (10)	0.0070 (9)	0.0049 (8)	−0.0021 (8)
C7	0.0245 (10)	0.0297 (10)	0.0274 (10)	0.0006 (8)	0.0091 (8)	−0.0041 (8)
C8	0.0262 (10)	0.0315 (10)	0.0278 (10)	0.0012 (8)	0.0126 (8)	0.0004 (8)
C9	0.0251 (10)	0.0306 (10)	0.0235 (9)	0.0069 (8)	0.0039 (7)	0.0033 (8)
C10	0.0325 (11)	0.0388 (12)	0.0324 (11)	0.0030 (9)	0.0064 (9)	−0.0075 (9)
C11	0.0321 (12)	0.0494 (14)	0.0525 (15)	−0.0054 (11)	0.0068 (11)	−0.0071 (12)
C12	0.0269 (11)	0.0633 (17)	0.0490 (14)	0.0002 (11)	0.0115 (10)	0.0019 (13)
C13	0.0363 (13)	0.0616 (16)	0.0379 (13)	0.0083 (12)	0.0149 (10)	−0.0040 (12)
C14	0.0333 (12)	0.0424 (13)	0.0305 (11)	0.0034 (9)	0.0065 (9)	−0.0061 (9)
C15	0.0391 (12)	0.0295 (11)	0.0416 (12)	−0.0029 (9)	0.0179 (10)	0.0002 (9)

Geometric parameters (Å, °)

Br—C4	1.902 (2)	C6—H6	0.9500
Br—O2^{i}	3.204 (2)	C8—C15	1.480 (3)
S—O2	1.492 (2)	C9—C10	1.380 (3)
S—C1	1.756 (2)	C9—C14	1.391 (3)
S—C9	1.796 (2)	C10—C11	1.389 (3)
O1—C8	1.373 (3)	C10—H10	0.9500
O1—C7	1.380 (2)	C11—C12	1.373 (4)

C1—C8	1.359 (3)	C11—H11	0.9500
C1—C2	1.447 (3)	C12—C13	1.384 (4)
C2—C7	1.397 (3)	C12—H12	0.9500
C2—C3	1.404 (3)	C13—C14	1.387 (4)
C3—C4	1.385 (3)	C13—H13	0.9500
C3—H3	0.9500	C14—H14	0.9500
C4—C5	1.393 (3)	C15—H15A	0.9800
C5—C6	1.387 (3)	C15—H15B	0.9800
C5—H5	0.9500	C15—H15C	0.9800
C6—C7	1.380 (3)		
O2—S—C1	109.34 (10)	C1—C8—C15	133.6 (2)
O2—S—C9	106.69 (10)	O1—C8—C15	115.56 (19)
C1—S—C9	98.07 (9)	C10—C9—C14	121.5 (2)
C8—O1—C7	106.61 (16)	C10—C9—S	119.09 (16)
C8—C1—C2	107.41 (17)	C14—C9—S	119.24 (17)
C8—C1—S	121.56 (16)	C9—C10—C11	119.0 (2)
C2—C1—S	131.03 (16)	C9—C10—H10	120.5
C7—C2—C3	118.86 (19)	C11—C10—H10	120.5
C7—C2—C1	104.62 (18)	C12—C11—C10	120.3 (2)
C3—C2—C1	136.51 (18)	C12—C11—H11	119.8
C4—C3—C2	116.67 (19)	C10—C11—H11	119.8
C4—C3—H3	121.7	C11—C12—C13	120.4 (2)
C2—C3—H3	121.7	C11—C12—H12	119.8
C3—C4—C5	123.5 (2)	C13—C12—H12	119.8
C3—C4—Br	119.49 (17)	C12—C13—C14	120.4 (2)
C5—C4—Br	116.97 (17)	C12—C13—H13	119.8
C6—C5—C4	120.2 (2)	C14—C13—H13	119.8
C6—C5—H5	119.9	C13—C14—C9	118.5 (2)
C4—C5—H5	119.9	C13—C14—H14	120.8
C7—C6—C5	116.33 (19)	C9—C14—H14	120.8
C7—C6—H6	121.8	C8—C15—H15A	109.5
C5—C6—H6	121.8	C8—C15—H15B	109.5
O1—C7—C6	125.09 (19)	H15A—C15—H15B	109.5
O1—C7—C2	110.53 (18)	C8—C15—H15C	109.5
C6—C7—C2	124.4 (2)	H15A—C15—H15C	109.5
C1—C8—O1	110.82 (18)	H15B—C15—H15C	109.5
O2—S—C1—C8	123.53 (18)	C3—C2—C7—C6	−0.7 (3)
C9—S—C1—C8	−125.54 (18)	C1—C2—C7—C6	−179.76 (19)
O2—S—C1—C2	−55.9 (2)	C2—C1—C8—O1	−1.0 (2)
C9—S—C1—C2	55.0 (2)	S—C1—C8—O1	179.47 (14)
C8—C1—C2—C7	0.7 (2)	C2—C1—C8—C15	179.1 (2)
S—C1—C2—C7	−179.84 (16)	S—C1—C8—C15	−0.4 (3)
C8—C1—C2—C3	−178.1 (2)	C7—O1—C8—C1	0.9 (2)
S—C1—C2—C3	1.4 (4)	C7—O1—C8—C15	−179.20 (18)
C7—C2—C3—C4	1.8 (3)	O2—S—C9—C10	−5.4 (2)
C1—C2—C3—C4	−179.6 (2)	C1—S—C9—C10	−118.41 (18)
C2—C3—C4—C5	−1.2 (3)	O2—S—C9—C14	179.50 (17)
C2—C3—C4—Br	177.61 (15)	C1—S—C9—C14	66.44 (19)

C3—C4—C5—C6	−0.6 (4)	C14—C9—C10—C11	0.1 (3)
Br—C4—C5—C6	−179.43 (17)	S—C9—C10—C11	−174.97 (19)
C4—C5—C6—C7	1.7 (3)	C9—C10—C11—C12	−0.3 (4)
C8—O1—C7—C6	179.18 (19)	C10—C11—C12—C13	−0.1 (4)
C8—O1—C7—C2	−0.4 (2)	C11—C12—C13—C14	0.7 (4)
C5—C6—C7—O1	179.39 (19)	C12—C13—C14—C9	−1.0 (4)
C5—C6—C7—C2	−1.0 (3)	C10—C9—C14—C13	0.6 (3)
C3—C2—C7—O1	178.89 (17)	S—C9—C14—C13	175.62 (18)
C1—C2—C7—O1	−0.1 (2)		

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*
C5—H5···O1^{ii}	0.95	2.49	3.420 (3)	166

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

Fig. 1

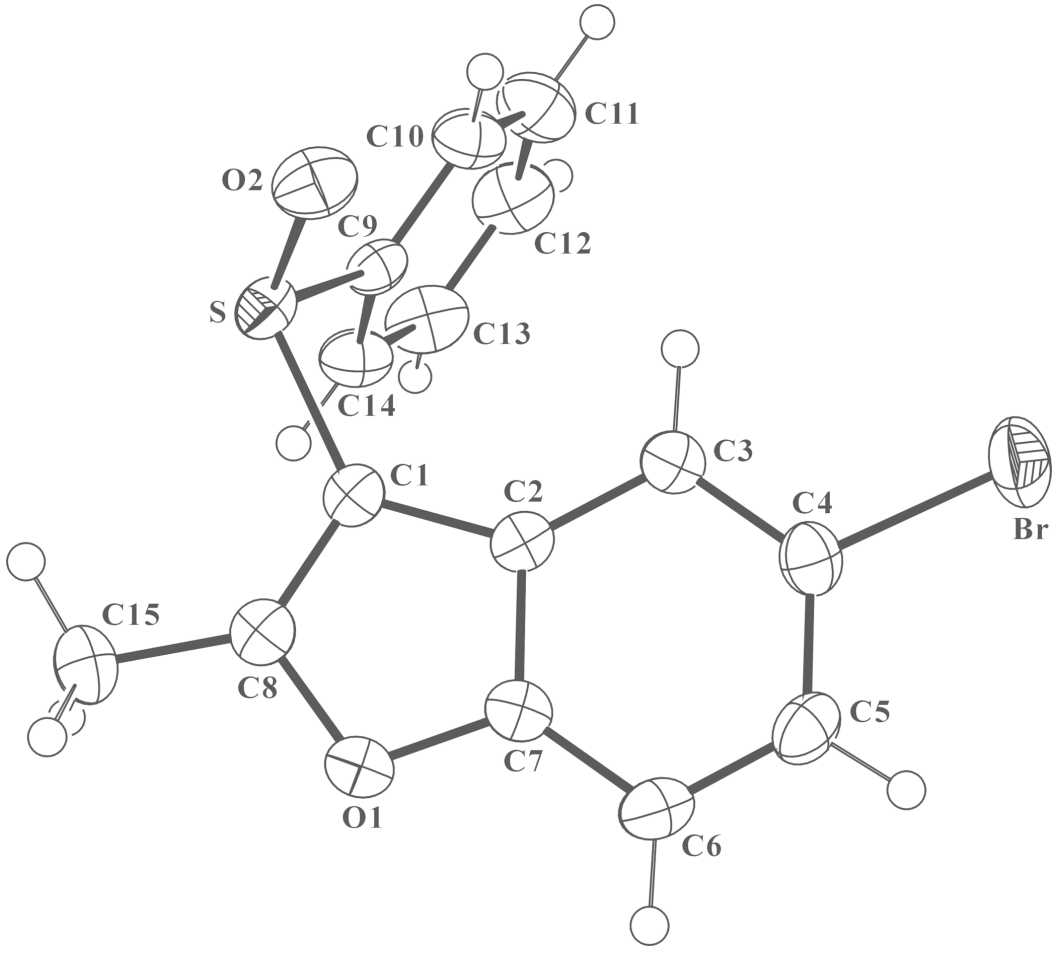
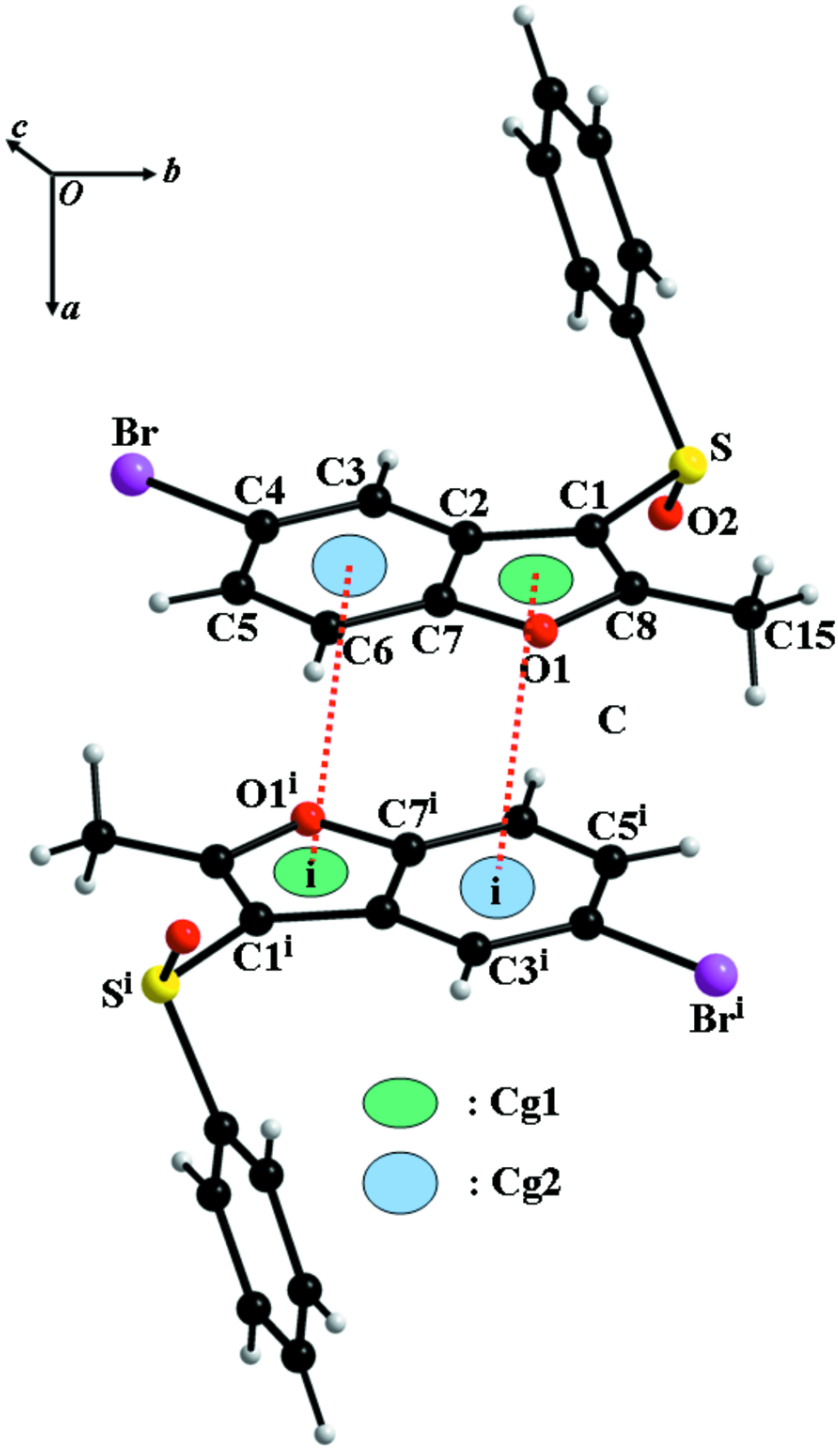


Fig. 2


c
b
O
a
Br
C4
C3
C2
C1
S
O2
C5
C6
C7
O1
C8
C15
C
O1i
C7i
C5i
i
i
C1i
S i
C3i
Bri
: Cg1
: Cg2