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2-(4-Bromophenyl)-5-methyl-3-methylsulfinyl-1-benzofuran

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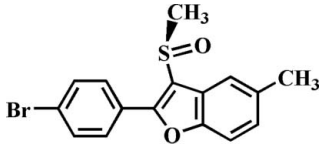
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.020; wR factor = 0.056; data-to-parameter ratio = 14.8.

The title compound, $C_{16}H_{13}BrO_2S$, was prepared by the oxidation of 2-(4-bromophenyl)-5-methyl-3-methylsulfanyl-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 4-bromophenyl ring is rotated out of the benzofuran plane with a dihedral angle of 26.21 (5)°. The crystal structure contains aromatic π–π interactions, with a centroid–centroid distance of 3.857 (2) Å between furan rings of neighboring molecules, and close CH_2—H$\cdots\pi$ contacts between the methylsulfinyl group and the aromatic system of the 4-bromophenyl ring.

Related literature

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



Experimental

Crystal data

$C_{16}H_{13}BrO_2S$
M_r = 349.23
Triclinic, $P\overline{1}$
a = 7.9693 (5) Å
b = 8.2016 (5) Å
c = 12.1816 (8) Å
α = 77.634 (1)°
β = 72.344 (1)°
γ = 68.777 (1)°
V = 702.34 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 3.07 mm^{-1}
T = 173 (2) K
0.40 × 0.40 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1999)
T_{min} = 0.297, T_{max} = 0.730
5542 measured reflections
2707 independent reflections
2576 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.056
S = 1.06
2707 reflections
183 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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

*Cg*2 is the centroid of the C9–C14 benzene ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C16—H16*B*$\cdots$*Cg*2^{i}	0.98	3.50	3.896 (2)	107

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

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: ZL2040).

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supplementary materials

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2-(4-Bromophenyl)-5-methyl-3-methylsulfinyl-1-benzofuran

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Comment

This work is related to preceding communications on the synthesis and structure of 5-methyl-3-methylsulfinyl-1-benzofuran analogues (Choi *et al.*, 2007*a*,b). 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 0.010 Å from the least-squares plane defined by the nine constituent atoms. The dihedral angle in (I) between the planes of the benzofuran and the 4-bromophenyl rings is 26.21 (5)°. The molecular packing (Fig. 2) is stabilized by π···π stacking interactions between adjacent furan units and the $Cg1\cdots Cg1^{ii}$ distance is 3.857 (2) Å. The molecular packing is further stabilized by CH_2—H···π interactions between the 3-methyl group and the 4-bromophenyl ring, with a C16—H16B···$Cg2^{i}$ separation of 3.50 Å. (Fig. 2 and hydrogen bonding table) (*Cg*1 and *Cg*2 are of the centroids of the O1/C8/C1/C2/C7 furan ring and the C9—C14 benzene ring, respectively; symmetry code as in Fig. 2).

Experimental

3-Chloroperbenzoic acid (77%, 247 mg, 1.10 mmol) was added in small portions to a stirred solution of 2-(4-bromophenyl)-5-methyl-3-methylsulfanyl-1-benzofuran (331 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 (hexane-ethyl acetate, 1:1 *v*/*v*) to afford the title compound as a colorless solid [yield 82%, m.p. 454–455 K; R_f = 0.31 (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 tetrahydrofuran at room temperature.

Refinement

All H atoms were geometrically located in ideal positions and refined using a riding model, with C—H = 0.95 Å for aromatic H atoms and C—H = 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 highest peak in the difference map is 1.16 Å from S and the largest hole is 0.58 Å from the S atom.

Figures

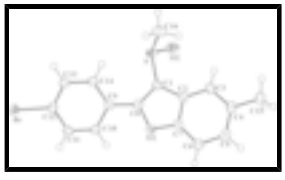


Fig. 1. The molecular structure of (I), showing displacement ellipsoides drawn at the 50% probability level.

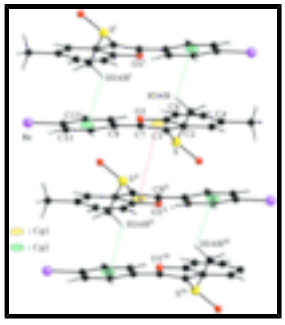


Fig. 2. π···π and CH_2—H···π interactions (dotted lines) in (I). *Cg* denotes the ring centroids. [Symmetry code: (i) 1 − *x*, 1 − *y*, 1 − *z*; (ii) 2 − *x*, 1 − *y*, 1 − *z*; (iii) 1 + *x*, *y*, *z*.]

2-(4-Bromophenyl)-5-methyl-3-methylsulfinyl-1-benzofuran

Crystal data

$C_{16}H_{13}BrO_2S$	$Z = 2$
$M_r = 349.23$	$F_{000} = 352$
Triclinic, $P\overline{1}$	$D_x = 1.651$ Mg m^{-3}
Hall symbol: -p 1	Melting point: 455 K
$a = 7.9693$ (5) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 8.2016$ (5) Å	Cell parameters from 4752 reflections
$c = 12.1816$ (8) Å	$\theta = 2.7$–28.3º
$\alpha = 77.634$ (1)º	$\mu = 3.07$ mm^{-1}
$\beta = 72.344$ (1)º	$T = 173$ (2) K
$\gamma = 68.777$ (1)º	Block, colorless
$V = 702.34$ (8) Å^3	0.40 × 0.40 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer	2707 independent reflections
Radiation source: fine-focus sealed tube	2576 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.014$
Detector resolution: 10.00 pixels mm^{-1}	$\theta_{max} = 26.0$º
$T = 173(2)$ K	$\theta_{min} = 1.8$º
φ and ω scans	$h = -9 \rightarrow 9$
Absorption correction: multi-scan (SADABS; Sheldrick, 1999)	$k = -10 \rightarrow 10$
$T_{min} = 0.297$, $T_{max} = 0.730$	$l = -15 \rightarrow 15$
5542 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
$R[F^2 > 2\sigma(F^2)] = 0.020$	H-atom parameters constrained

$wR(F^2) = 0.056$	$w = 1/[\sigma^2(F_o^2) + (0.031P)^2 + 0.2818P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
2707 reflections	$\Delta\rho_{max} = 0.27$ e Å^{-3}
183 parameters	$\Delta\rho_{min} = -0.32$ 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}
Br	0.67916 (2)	0.76665 (2)	−0.014702 (14)	0.02974 (7)
S	0.62961 (6)	0.79742 (5)	0.59939 (4)	0.02538 (10)
O1	0.84540 (16)	0.31832 (15)	0.49907 (10)	0.0242 (3)
O2	0.71676 (19)	0.82196 (18)	0.68449 (13)	0.0371 (3)
C1	0.7157 (2)	0.5700 (2)	0.58281 (15)	0.0228 (3)
C2	0.7688 (2)	0.4268 (2)	0.67133 (15)	0.0229 (3)
C3	0.7545 (2)	0.4103 (2)	0.79027 (15)	0.0261 (4)
H3	0.6988	0.5109	0.8312	0.031*
C4	0.8235 (2)	0.2437 (3)	0.84743 (15)	0.0278 (4)
C5	0.9052 (3)	0.0964 (2)	0.78581 (16)	0.0296 (4)
H5	0.9525	−0.0162	0.8262	0.036*
C6	0.9195 (2)	0.1090 (2)	0.66804 (16)	0.0277 (4)
H6	0.9742	0.0086	0.6270	0.033*
C7	0.8495 (2)	0.2764 (2)	0.61426 (15)	0.0237 (3)
C8	0.7634 (2)	0.4992 (2)	0.48187 (15)	0.0227 (3)
C9	0.7438 (2)	0.5682 (2)	0.36426 (14)	0.0221 (3)
C10	0.8668 (2)	0.4757 (2)	0.27141 (15)	0.0238 (3)
H10	0.9648	0.3717	0.2854	0.029*
C11	0.8462 (2)	0.5348 (2)	0.16032 (15)	0.0251 (4)
H11	0.9288	0.4711	0.0978	0.030*
C12	0.7041 (2)	0.6881 (2)	0.13980 (15)	0.0230 (3)
C13	0.5815 (2)	0.7830 (2)	0.22925 (16)	0.0259 (4)
H13	0.4858	0.8884	0.2142	0.031*
C14	0.6013 (2)	0.7212 (2)	0.34139 (16)	0.0262 (4)
H14	0.5165	0.7840	0.4037	0.031*

C15	0.8122 (3)	0.2222 (3)	0.97538 (17)	0.0355 (4)
H15A	0.9345	0.2053	0.9864	0.053*
H15B	0.7729	0.1194	1.0137	0.053*
H15C	0.7219	0.3279	1.0092	0.053*
C16	0.3960 (3)	0.8089 (3)	0.67703 (19)	0.0355 (4)
H16A	0.3976	0.7243	0.7473	0.053*
H16B	0.3358	0.7806	0.6277	0.053*
H16C	0.3268	0.9282	0.6985	0.053*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.03109 (11)	0.02932 (11)	0.02304 (11)	−0.00261 (8)	−0.00838 (7)	−0.00132 (7)
S	0.0259 (2)	0.0197 (2)	0.0294 (2)	−0.00610 (17)	−0.00501 (17)	−0.00528 (16)
O1	0.0280 (6)	0.0201 (6)	0.0225 (6)	−0.0053 (5)	−0.0061 (5)	−0.0028 (5)
O2	0.0359 (7)	0.0311 (7)	0.0504 (9)	−0.0064 (6)	−0.0166 (6)	−0.0162 (6)
C1	0.0236 (8)	0.0201 (8)	0.0242 (8)	−0.0060 (6)	−0.0061 (7)	−0.0032 (6)
C2	0.0230 (8)	0.0224 (8)	0.0247 (9)	−0.0088 (7)	−0.0060 (7)	−0.0026 (7)
C3	0.0283 (9)	0.0278 (9)	0.0250 (9)	−0.0109 (7)	−0.0072 (7)	−0.0045 (7)
C4	0.0283 (9)	0.0346 (10)	0.0240 (9)	−0.0142 (7)	−0.0093 (7)	0.0009 (7)
C5	0.0287 (9)	0.0270 (9)	0.0319 (10)	−0.0090 (7)	−0.0108 (8)	0.0042 (7)
C6	0.0281 (9)	0.0236 (9)	0.0300 (9)	−0.0064 (7)	−0.0070 (7)	−0.0035 (7)
C7	0.0236 (8)	0.0262 (9)	0.0219 (8)	−0.0088 (7)	−0.0052 (7)	−0.0032 (7)
C8	0.0214 (8)	0.0200 (8)	0.0251 (9)	−0.0055 (6)	−0.0051 (7)	−0.0023 (6)
C9	0.0226 (8)	0.0227 (8)	0.0221 (8)	−0.0092 (7)	−0.0043 (6)	−0.0032 (6)
C10	0.0204 (8)	0.0206 (8)	0.0265 (9)	−0.0039 (6)	−0.0035 (7)	−0.0027 (7)
C11	0.0231 (8)	0.0245 (9)	0.0226 (8)	−0.0052 (7)	−0.0001 (7)	−0.0048 (7)
C12	0.0232 (8)	0.0239 (8)	0.0223 (8)	−0.0085 (7)	−0.0060 (6)	−0.0009 (6)
C13	0.0218 (8)	0.0246 (9)	0.0288 (9)	−0.0023 (7)	−0.0077 (7)	−0.0050 (7)
C14	0.0232 (8)	0.0265 (9)	0.0262 (9)	−0.0041 (7)	−0.0037 (7)	−0.0079 (7)
C15	0.0424 (11)	0.0398 (11)	0.0275 (10)	−0.0151 (9)	−0.0149 (8)	0.0020 (8)
C16	0.0260 (9)	0.0343 (10)	0.0437 (11)	−0.0071 (8)	−0.0022 (8)	−0.0133 (9)

Geometric parameters (Å, °)

Br—C12	1.900 (2)	C8—C9	1.457 (2)
S—O2	1.492 (1)	C9—C14	1.398 (2)
S—C1	1.773 (2)	C9—C10	1.404 (2)
S—C16	1.793 (2)	C10—C11	1.375 (2)
O1—C7	1.379 (2)	C10—H10	0.9500
O1—C8	1.385 (2)	C11—C12	1.390 (2)
C1—C8	1.362 (2)	C11—H11	0.9500
C1—C2	1.447 (2)	C12—C13	1.384 (2)
C2—C7	1.393 (2)	C13—C14	1.387 (3)
C2—C3	1.399 (2)	C13—H13	0.9500
C3—C4	1.391 (3)	C14—H14	0.9500
C3—H3	0.9500	C15—H15A	0.9800
C4—C5	1.403 (3)	C15—H15B	0.9800
C4—C15	1.509 (2)	C15—H15C	0.9800

C5—C6	1.388 (3)	C16—H16A	0.9800
C5—H5	0.9500	C16—H16B	0.9800
C6—C7	1.381 (2)	C16—H16C	0.9800
C6—H6	0.9500		
O2—S—C1	107.10 (8)	C14—C9—C8	121.74 (15)
O2—S—C16	105.85 (9)	C10—C9—C8	119.43 (15)
C1—S—C16	97.86 (9)	C11—C10—C9	120.35 (15)
C7—O1—C8	106.59 (13)	C11—C10—H10	119.8
C8—C1—C2	107.47 (15)	C9—C10—H10	119.8
C8—C1—S	126.27 (13)	C10—C11—C12	119.72 (15)
C2—C1—S	125.88 (13)	C10—C11—H11	120.1
C7—C2—C3	119.17 (16)	C12—C11—H11	120.1
C7—C2—C1	104.94 (15)	C13—C12—C11	121.34 (16)
C3—C2—C1	135.89 (16)	C13—C12—Br	119.91 (13)
C4—C3—C2	118.69 (16)	C11—C12—Br	118.76 (13)
C4—C3—H3	120.7	C12—C13—C14	118.67 (16)
C2—C3—H3	120.7	C12—C13—H13	120.7
C3—C4—C5	119.98 (16)	C14—C13—H13	120.7
C3—C4—C15	119.86 (17)	C13—C14—C9	121.12 (16)
C5—C4—C15	120.16 (17)	C13—C14—H14	119.4
C6—C5—C4	122.46 (17)	C9—C14—H14	119.4
C6—C5—H5	118.8	C4—C15—H15A	109.5
C4—C5—H5	118.8	C4—C15—H15B	109.5
C7—C6—C5	115.92 (16)	H15A—C15—H15B	109.5
C7—C6—H6	122.0	C4—C15—H15C	109.5
C5—C6—H6	122.0	H15A—C15—H15C	109.5
O1—C7—C6	125.55 (15)	H15B—C15—H15C	109.5
O1—C7—C2	110.66 (15)	S—C16—H16A	109.5
C6—C7—C2	123.78 (16)	S—C16—H16B	109.5
C1—C8—O1	110.33 (15)	H16A—C16—H16B	109.5
C1—C8—C9	135.14 (16)	S—C16—H16C	109.5
O1—C8—C9	114.51 (14)	H16A—C16—H16C	109.5
C14—C9—C10	118.78 (16)	H16B—C16—H16C	109.5
O2—S—C1—C8	−140.78 (16)	C1—C2—C7—C6	−179.89 (16)
C16—S—C1—C8	109.89 (17)	C2—C1—C8—O1	−0.28 (19)
O2—S—C1—C2	31.28 (17)	S—C1—C8—O1	172.99 (12)
C16—S—C1—C2	−78.04 (16)	C2—C1—C8—C9	177.77 (18)
C8—C1—C2—C7	0.86 (18)	S—C1—C8—C9	−9.0 (3)
S—C1—C2—C7	−172.44 (13)	C7—O1—C8—C1	−0.43 (18)
C8—C1—C2—C3	−178.08 (19)	C7—O1—C8—C9	−178.91 (14)
S—C1—C2—C3	8.6 (3)	C1—C8—C9—C14	−26.6 (3)
C7—C2—C3—C4	0.7 (2)	O1—C8—C9—C14	151.41 (16)
C1—C2—C3—C4	179.51 (18)	C1—C8—C9—C10	155.75 (19)
C2—C3—C4—C5	−0.1 (3)	O1—C8—C9—C10	−26.3 (2)
C2—C3—C4—C15	179.24 (16)	C14—C9—C10—C11	−0.3 (3)
C3—C4—C5—C6	−0.5 (3)	C8—C9—C10—C11	177.44 (15)
C15—C4—C5—C6	−179.85 (17)	C9—C10—C11—C12	0.8 (3)
C4—C5—C6—C7	0.5 (3)	C10—C11—C12—C13	−0.2 (3)

C8—O1—C7—C6	179.72 (17)	C10—C11—C12—Br	179.97 (13)
C8—O1—C7—C2	1.00 (18)	C11—C12—C13—C14	−0.7 (3)
C5—C6—C7—O1	−178.41 (16)	Br—C12—C13—C14	179.05 (13)
C5—C6—C7—C2	0.1 (3)	C12—C13—C14—C9	1.2 (3)
C3—C2—C7—O1	178.01 (14)	C10—C9—C14—C13	−0.7 (3)
C1—C2—C7—O1	−1.15 (18)	C8—C9—C14—C13	−178.39 (16)
C3—C2—C7—C6	−0.7 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16B···Cg2^{i}	0.98	3.50	3.896 (2)	107

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

Fig. 1

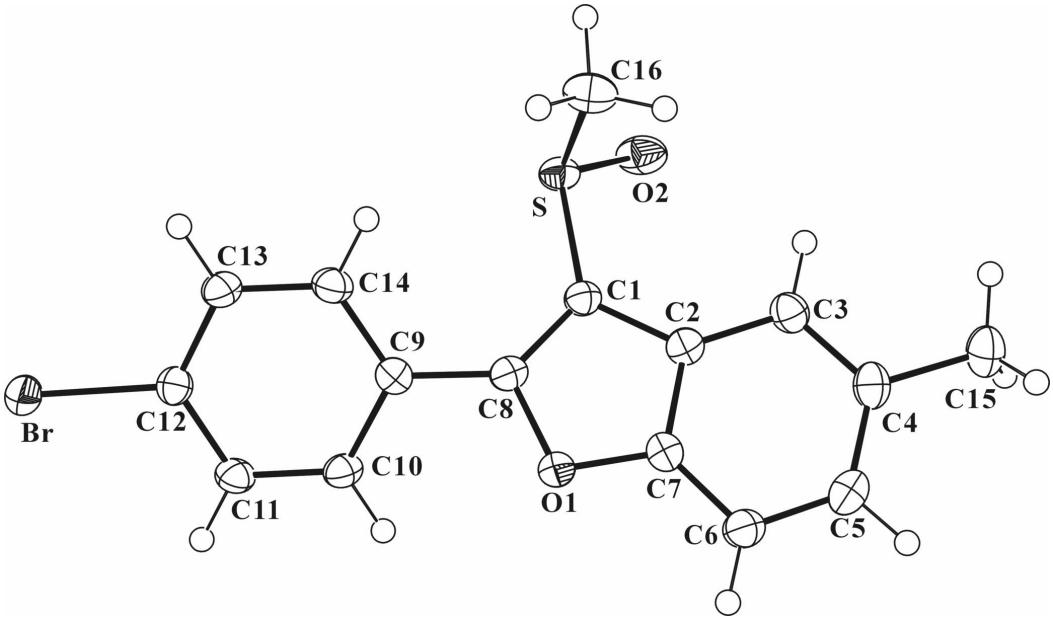
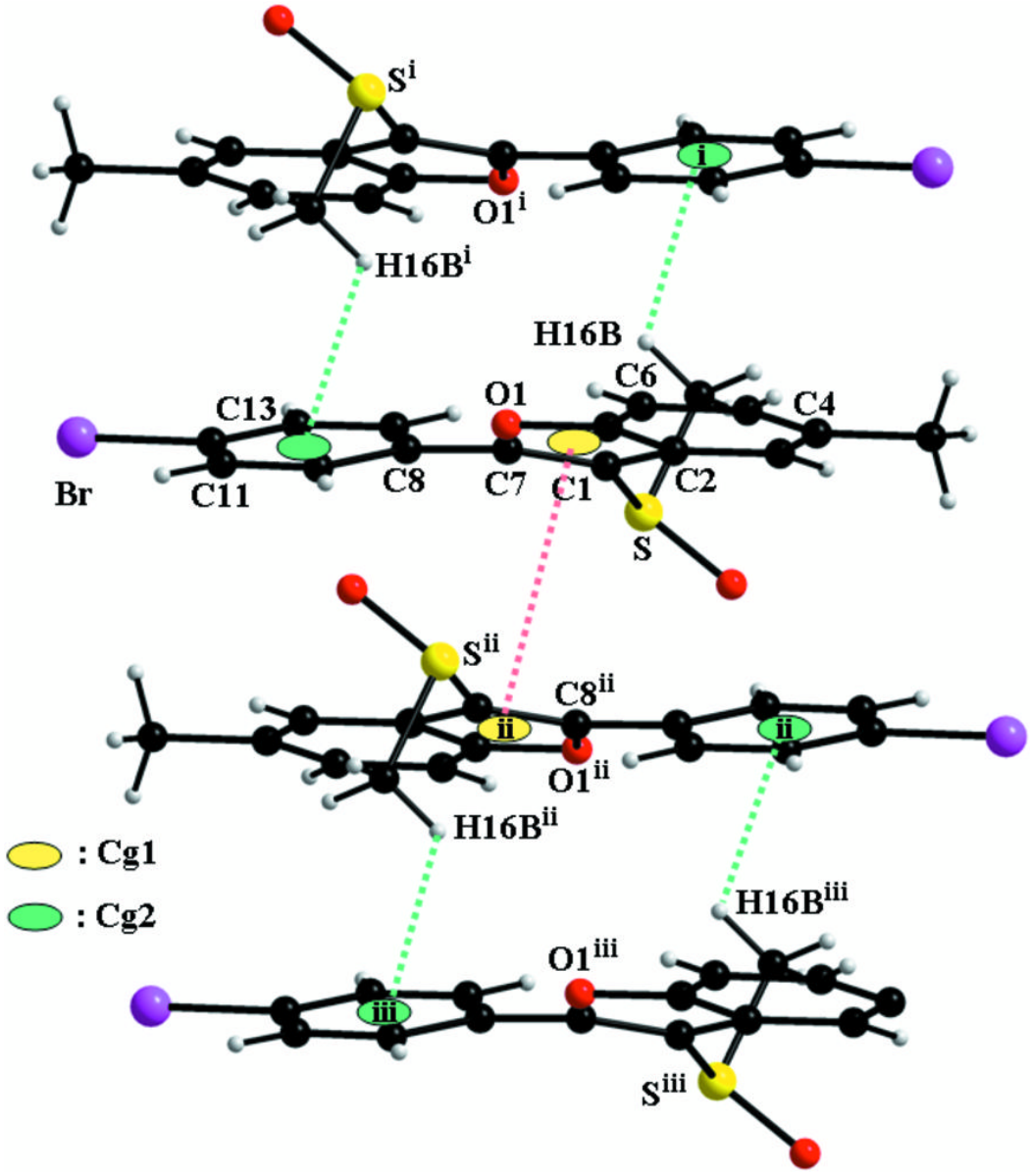


Fig. 2


S^i
O1^i
H16B^i
i
H16B
C6
C4
O1
C13
Br
C11
C8
C7
C1
C2
S
S^ii
C8^ii
ii
ii
O1^ii
H16B^ii
: Cg1
: Cg2
H16B^iii
O1^iii
iii
S^iii