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2-(4-Bromophenyl)-5,7-dimethyl-3-methylsulfanyl-1-benzofuran

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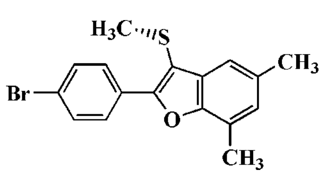
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.026; wR factor = 0.066; data-to-parameter ratio = 18.9.

The title compound, $C_{17}H_{15}BrOS$, was prepared by the Lewis acid-catalysed reaction of 2,4-dimethylphenol with 4′-bromo-2-chloro-2-(methylsulfanyl)acetophenone. The 4-bromophenyl ring is rotated slightly out of the benzofuran plane, making a dihedral angle of 8.4 (1)°. The crystal structure is stabilized by a CH_2—H$\cdots\pi$ interaction between the 5-methyl group and the benzene ring of the benzofuran system.

Related literature

For the crystal structures of similar 2-(4-bromophenyl)-1-benzofuran compounds, see: Choi *et al.* (2007*a*,*b*).



Experimental

Crystal data

$C_{17}H_{15}BrOS$
M_r = 347.26
Monoclinic, $P2_1$
a = 5.2332 (1) Å
b = 10.6602 (2) Å
c = 13.6374 (2) Å
β = 98.092 (1)°
V = 753.21 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.86 mm^{-1}
T = 296 (2) K
0.30 × 0.24 × 0.08 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
T_{min} = 0.481, T_{max} = 0.804
7779 measured reflections
3448 independent reflections
2957 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.066
S = 0.87
3448 reflections
182 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Absolute structure: Flack (1983), 1477 Friedel pairs
Flack parameter: 0.011 (7)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C15—H15$A\cdots Cg^{i}$	0.96	2.97	3.891 (3)	161

Symmetry code: (i) $x - 1, y, z$. Cg is the centroid of the C2–C7 benzene ring.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); 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: OM2192).

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supplementary materials

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2-(4-Bromophenyl)-5,7-dimethyl-3-methylsulfanyl-1-benzofuran

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Comment

As part of our continuing studies on the synthesis and structures of 2-(4-bromophenyl)-1-benzofuran derivatives, the crystal structures of 2-(4-bromophenyl)-5-methyl-3-methylsulfinyl-1-benzofuran (Choi *et al.*, 2007*a*) and 2-(4-bromophenyl)-5,7-dimethyl-3-methylsulfinyl-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, 2-(4-bromophenyl)-5,7-dimethyl-3-methylsulfanyl-1-benzofuran (Fig. 1).

The benzofuran unit is essentially planar, with a mean deviation of 0.007 Å from the least-squares plane defined by the nine constituent atoms. The dihedral angle in the title compound formed by the plane of the benzofuran unit and the plane of the 4-bromophenyl ring is 8.4 (1)°. The molecular packing (Fig. 2) is stabilized by a CH_2—H···π interaction between 5-methyl group and the benzene ring of benzofuran system, with a C15—H15A···Cg^{i} separation of 2.97 Å (Table 1; *Cg* is a centroid of the C2—C7 benzene ring, symmetry code as in Fig. 2).

Experimental

Zinc chloride (546 mg, 4.0 mmol) was added at room temperature to a stirred solution of 2,4-dimethylphenol (489 mg, 4.0 mmol) and 4'-bromo-2-chloro-2-(methylsulfanyl)acetophenone (1.12 g, 4.0 mmol) in dichloromethane (30 ml) and stirred for 40 min. The mixture was quenched with water and the organic layer was separated, dried over magnesium sulfate, filtered and concentrated in vacuum. The residue was purified by column chromatography (CCl_4) to afford the title compound as a colorless solid [yield 52%, m.p. 385–386 K; R_f = 0.81 (CCl_4)]. 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 geometrically located in ideal positions 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) = 1.2U_{eq}(C) for aromatic H atoms, and 1.5U_{eq}(C) for methyl H atoms.

Figures

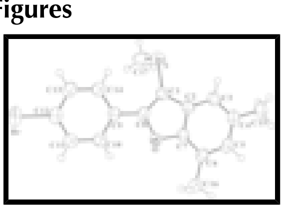


Fig. 1. The molecular structure of the title compound. showing displacement ellipsoids drawn at the 50% probability level.

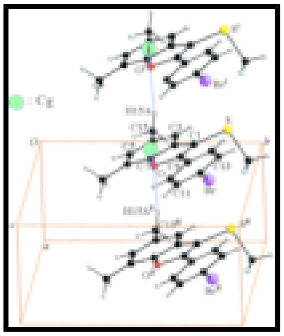


Fig. 2. C—H···π interaction (dotted lines) in the title compound. *Cg* denotes ring centroid. [Symmetry code: (i) $x - 1, y, z$; (ii) $x + 1, y, z$.]

2-(4-Bromophenyl)-5,7-dimethyl-3-methylsulfanyl-1-benzofuran

Crystal data

$C_{17}H_{15}BrOS$	$F_{000} = 352$
$M_r = 347.26$	$D_x = 1.531$ Mg m^{-3}
Monoclinic, $P2_1$	Melting point: 385-386 K
Hall symbol: p 2yb	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 5.2332$ (1) Å	Cell parameters from 3988 reflections
$b = 10.6602$ (2) Å	$\theta = 3.0–27.1°$
$c = 13.6374$ (2) Å	$\mu = 2.86$ mm^{-1}
$\beta = 98.092$ (1)°	$T = 296$ (2) K
$V = 753.21$ (2) Å^3	Block, silver
$Z = 2$	0.30 × 0.24 × 0.08 mm

Data collection

Bruker SMART CCD diffractometer	3448 independent reflections
Radiation source: fine-focus sealed tube	2957 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.020$
Detector resolution: 10.0 pixels mm^{-1}	$\theta_{max} = 28.4°$
$T = 296(2)$ K	$\theta_{min} = 1.5°$
φ and ω scans	$h = -6 \rightarrow 6$
Absorption correction: multi-scan (SADABS; Sheldrick, 2000)	$k = -14 \rightarrow 14$
$T_{min} = 0.481$, $T_{max} = 0.804$	$l = -17 \rightarrow 18$
7779 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.026$	$w = 1/[\sigma^2(F_o^2) + (0.0308P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$wR(F^2)$ = 0.066	$(\Delta/\sigma)_{max}$ = 0.001
S = 0.87	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
3448 reflections	$\Delta\rho_{min}$ = −0.20 e Å^{-3}
182 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), 1477 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.011 (7)
Secondary atom site location: difference Fourier map	

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	0.53020 (5)	0.73216 (4)	−0.179068 (16)	0.06373 (10)
S	−0.30384 (12)	0.86959 (6)	0.21002 (5)	0.05180 (16)
O	0.0436 (3)	0.53588 (14)	0.23230 (12)	0.0412 (4)
C1	−0.1770 (4)	0.7187 (3)	0.23497 (15)	0.0404 (5)
C2	−0.2370 (5)	0.6414 (2)	0.31587 (17)	0.0407 (5)
C3	−0.3917 (5)	0.6547 (2)	0.39043 (19)	0.0470 (6)
H3	−0.4889	0.7271	0.3946	0.056*
C4	−0.3984 (5)	0.5590 (2)	0.45776 (18)	0.0455 (5)
C5	−0.2494 (5)	0.4511 (2)	0.44955 (17)	0.0450 (5)
H5	−0.2559	0.3878	0.4960	0.054*
C6	−0.0931 (4)	0.4327 (2)	0.37673 (17)	0.0410 (5)
C7	−0.0959 (4)	0.5314 (2)	0.31082 (17)	0.0398 (5)
C8	−0.0091 (4)	0.6519 (2)	0.18701 (16)	0.0404 (5)
C9	0.1192 (5)	0.6724 (2)	0.10053 (18)	0.0404 (5)
C10	0.3069 (5)	0.5879 (2)	0.07758 (18)	0.0468 (5)
H10	0.3491	0.5187	0.1182	0.056*
C11	0.4310 (5)	0.6059 (2)	−0.00479 (19)	0.0510 (6)
H11	0.5568	0.5498	−0.0190	0.061*
C12	0.3659 (5)	0.7077 (2)	−0.06535 (17)	0.0465 (6)
C13	0.1811 (5)	0.7927 (3)	−0.04460 (19)	0.0527 (6)
H13	0.1395	0.8613	−0.0859	0.063*
C14	0.0585 (5)	0.7753 (2)	0.03788 (19)	0.0491 (6)
H14	−0.0655	0.8326	0.0518	0.059*
C15	−0.5633 (5)	0.5692 (3)	0.5390 (2)	0.0598 (7)

H15A	−0.7403	0.5805	0.5105	0.072*
H15B	−0.5081	0.6398	0.5803	0.072*
H15C	−0.5471	0.4940	0.5780	0.072*
C16	0.0696 (5)	0.3184 (2)	0.3687 (2)	0.0533 (6)
H16A	0.0178	0.2785	0.3060	0.080*
H16B	0.0473	0.2611	0.4212	0.080*
H16C	0.2478	0.3424	0.3739	0.080*
C17	−0.0310 (6)	0.9643 (3)	0.2543 (3)	0.0748 (10)
H17A	−0.0756	1.0512	0.2448	0.112*
H17B	0.1087	0.9444	0.2183	0.112*
H17C	0.0207	0.9483	0.3235	0.112*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.06774 (17)	0.0829 (2)	0.04418 (13)	−0.01530 (17)	0.02044 (10)	0.00117 (15)
S	0.0504 (4)	0.0453 (3)	0.0605 (4)	0.0108 (3)	0.0106 (3)	0.0058 (3)
O	0.0507 (9)	0.0368 (9)	0.0387 (9)	0.0033 (7)	0.0148 (7)	0.0012 (6)
C1	0.0428 (11)	0.0384 (12)	0.0404 (10)	0.0022 (11)	0.0073 (8)	−0.0004 (11)
C2	0.0446 (12)	0.0388 (12)	0.0397 (12)	−0.0037 (10)	0.0093 (9)	−0.0049 (9)
C3	0.0501 (13)	0.0448 (13)	0.0483 (14)	0.0014 (11)	0.0144 (11)	−0.0066 (11)
C4	0.0486 (12)	0.0477 (14)	0.0419 (13)	−0.0117 (10)	0.0125 (10)	−0.0094 (10)
C5	0.0538 (14)	0.0469 (13)	0.0354 (11)	−0.0120 (11)	0.0099 (10)	0.0023 (10)
C6	0.0457 (13)	0.0378 (12)	0.0395 (12)	−0.0050 (10)	0.0061 (10)	−0.0001 (10)
C7	0.0441 (12)	0.0418 (13)	0.0347 (11)	−0.0027 (11)	0.0097 (9)	−0.0039 (9)
C8	0.0456 (13)	0.0394 (12)	0.0361 (12)	−0.0026 (10)	0.0059 (10)	0.0017 (9)
C9	0.0425 (12)	0.0419 (12)	0.0367 (12)	−0.0040 (10)	0.0055 (9)	−0.0017 (9)
C10	0.0548 (14)	0.0434 (13)	0.0438 (13)	0.0038 (11)	0.0120 (10)	0.0031 (11)
C11	0.0533 (15)	0.0524 (15)	0.0497 (14)	0.0072 (12)	0.0151 (11)	−0.0009 (12)
C12	0.0470 (12)	0.0553 (17)	0.0375 (11)	−0.0146 (12)	0.0071 (9)	−0.0032 (11)
C13	0.0625 (16)	0.0527 (14)	0.0427 (14)	−0.0017 (13)	0.0068 (12)	0.0084 (11)
C14	0.0528 (14)	0.0491 (15)	0.0461 (14)	0.0040 (11)	0.0089 (11)	0.0037 (10)
C15	0.0655 (17)	0.0646 (17)	0.0546 (16)	−0.0084 (14)	0.0272 (12)	−0.0055 (13)
C16	0.0633 (17)	0.0434 (14)	0.0561 (16)	0.0042 (12)	0.0183 (13)	0.0068 (11)
C17	0.069 (2)	0.0472 (17)	0.104 (3)	−0.0002 (14)	0.0003 (18)	−0.0102 (17)

Geometric parameters (Å, °)

Br—C12	1.894 (2)	C9—C14	1.399 (3)
S—C1	1.755 (3)	C10—C11	1.387 (4)
S—C17	1.783 (3)	C10—H10	0.9300
O—C7	1.379 (3)	C11—C12	1.377 (4)
O—C8	1.393 (3)	C11—H11	0.9300
C1—C8	1.367 (3)	C12—C13	1.383 (4)
C1—C2	1.447 (3)	C13—C14	1.384 (4)
C2—C3	1.393 (3)	C13—H13	0.9300
C2—C7	1.393 (3)	C14—H14	0.9300
C3—C4	1.377 (4)	C15—H15A	0.9600
C3—H3	0.9300	C15—H15B	0.9600

C4—C5	1.402 (3)	C15—H15C	0.9600
C4—C15	1.501 (3)	C16—H16A	0.9600
C5—C6	1.387 (4)	C16—H16B	0.9600
C5—H5	0.9300	C16—H16C	0.9600
C6—C7	1.382 (3)	C17—H17A	0.9600
C6—C16	1.499 (3)	C17—H17B	0.9600
C8—C9	1.453 (3)	C17—H17C	0.9600
C9—C10	1.401 (3)		

C1—S—C17	100.98 (13)	C9—C10—H10	119.5
C7—O—C8	106.38 (17)	C12—C11—C10	119.3 (2)
C8—C1—C2	106.9 (2)	C12—C11—H11	120.3
C8—C1—S	129.47 (19)	C10—C11—H11	120.3
C2—C1—S	123.58 (18)	C11—C12—C13	121.0 (2)
C3—C2—C7	119.2 (2)	C11—C12—Br	119.82 (19)
C3—C2—C1	135.2 (2)	C13—C12—Br	119.21 (19)
C7—C2—C1	105.6 (2)	C12—C13—C14	119.7 (2)
C4—C3—C2	118.9 (2)	C12—C13—H13	120.1
C4—C3—H3	120.6	C14—C13—H13	120.1
C2—C3—H3	120.6	C13—C14—C9	120.7 (2)
C3—C4—C5	119.2 (2)	C13—C14—H14	119.7
C3—C4—C15	120.6 (2)	C9—C14—H14	119.7
C5—C4—C15	120.2 (2)	C4—C15—H15A	109.5
C6—C5—C4	124.4 (2)	C4—C15—H15B	109.5
C6—C5—H5	117.8	H15A—C15—H15B	109.5
C4—C5—H5	117.8	C4—C15—H15C	109.5
C7—C6—C5	113.8 (2)	H15A—C15—H15C	109.5
C7—C6—C16	121.7 (2)	H15B—C15—H15C	109.5
C5—C6—C16	124.5 (2)	C6—C16—H16A	109.5
O—C7—C6	125.0 (2)	C6—C16—H16B	109.5
O—C7—C2	110.56 (19)	H16A—C16—H16B	109.5
C6—C7—C2	124.5 (2)	C6—C16—H16C	109.5
C1—C8—O	110.5 (2)	H16A—C16—H16C	109.5
C1—C8—C9	135.3 (2)	H16B—C16—H16C	109.5
O—C8—C9	114.1 (2)	S—C17—H17A	109.5
C10—C9—C14	118.3 (2)	S—C17—H17B	109.5
C10—C9—C8	120.2 (2)	H17A—C17—H17B	109.5
C14—C9—C8	121.5 (2)	S—C17—H17C	109.5
C11—C10—C9	121.0 (2)	H17A—C17—H17C	109.5
C11—C10—H10	119.5	H17B—C17—H17C	109.5

C17—S—C1—C8	−74.5 (3)	C3—C2—C7—C6	−1.3 (3)
C17—S—C1—C2	104.8 (2)	C1—C2—C7—C6	178.4 (2)
C8—C1—C2—C3	−180.0 (2)	C2—C1—C8—O	−0.1 (2)
S—C1—C2—C3	0.6 (4)	S—C1—C8—O	179.26 (16)
C8—C1—C2—C7	0.4 (2)	C2—C1—C8—C9	178.2 (2)
S—C1—C2—C7	−179.06 (16)	S—C1—C8—C9	−2.4 (4)
C7—C2—C3—C4	0.7 (3)	C7—O—C8—C1	−0.2 (2)
C1—C2—C3—C4	−178.9 (2)	C7—O—C8—C9	−178.89 (17)
C2—C3—C4—C5	0.1 (3)	C1—C8—C9—C10	172.7 (3)

C2—C3—C4—C15	−179.7 (2)	O—C8—C9—C10	−9.0 (3)
C3—C4—C5—C6	−0.4 (4)	C1—C8—C9—C14	−7.9 (4)
C15—C4—C5—C6	179.4 (2)	O—C8—C9—C14	170.44 (19)
C4—C5—C6—C7	−0.2 (3)	C14—C9—C10—C11	0.4 (3)
C4—C5—C6—C16	179.0 (2)	C8—C9—C10—C11	179.8 (2)
C8—O—C7—C6	−178.4 (2)	C9—C10—C11—C12	−0.7 (4)
C8—O—C7—C2	0.4 (2)	C10—C11—C12—C13	0.7 (4)
C5—C6—C7—O	179.7 (2)	C10—C11—C12—Br	−179.15 (18)
C16—C6—C7—O	0.5 (3)	C11—C12—C13—C14	−0.3 (4)
C5—C6—C7—C2	1.0 (3)	Br—C12—C13—C14	179.56 (18)
C16—C6—C7—C2	−178.2 (2)	C12—C13—C14—C9	−0.1 (4)
C3—C2—C7—O	179.8 (2)	C10—C9—C14—C13	0.1 (3)
C1—C2—C7—O	−0.5 (2)	C8—C9—C14—C13	−179.4 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15—H15A···Cg^{i}	0.96	2.97	3.891 (3)	161

Symmetry codes: (i) *x*−1, *y*, *z*.

Fig. 1

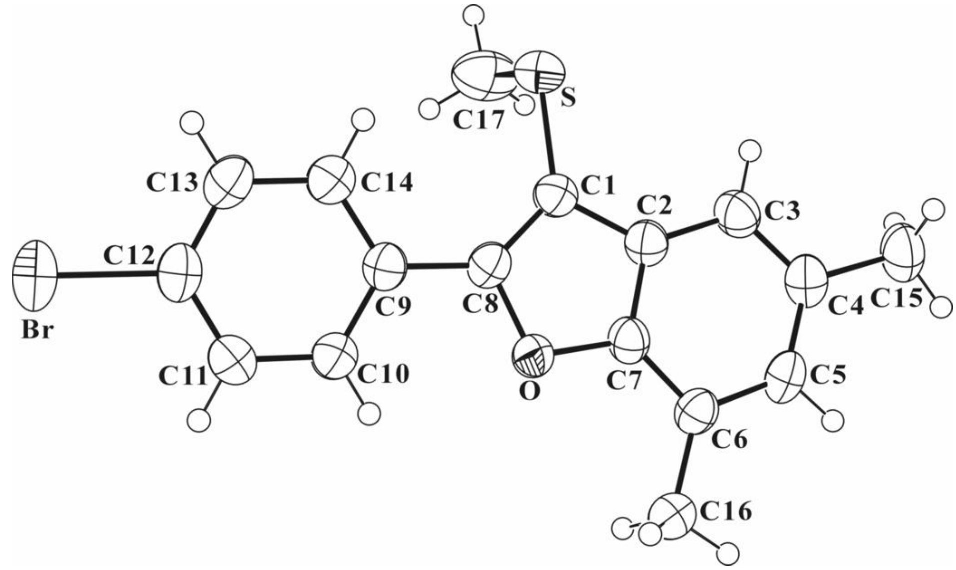
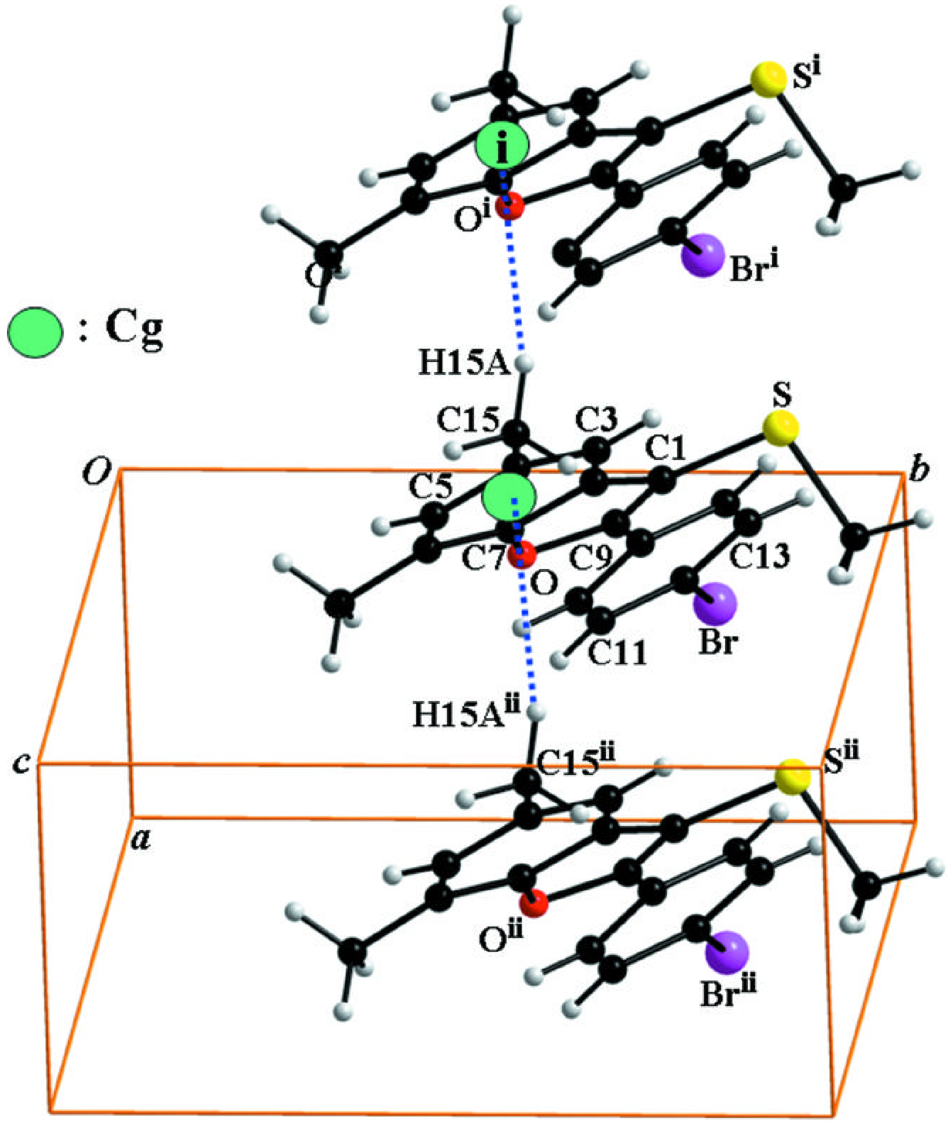


Fig. 2


Si
Oi
Bri
: Cg
H15A
C15
C3
C1
S
O
b
C5
C7
O
C9
C13
C11
Br
H15Aii
C15ii
Sii
c
a
Oii
Brii