

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

5-Bromo-3-(4-chlorophenylsulfinyl)-2-methyl-1-benzofuran

Hong Dae Choi,^a Pil Ja Seo,^a Byung Wha Son^b and Uk Lee^{b*}^aDepartment of Chemistry, Donggeui University, San 24 Kaya-dong Busanjin-gu, Busan 614-714, Republic of Korea, and ^bDepartment 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

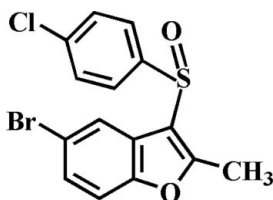
Received 28 September 2010; accepted 29 September 2010

Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.040; wR factor = 0.125; data-to-parameter ratio = 17.4.

In the title compound, $\text{C}_{15}\text{H}_{10}\text{BrClO}_2\text{S}$, the 4-chlorophenyl ring is oriented approximately perpendicular to the mean plane of the benzofuran ring [dihedral angle = $89.55(9)^\circ$]. In the crystal, molecules are linked through weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds and a $\text{Br}\cdots\text{Br}$ contact [3.783 (3) Å].

Related literature

For the biological 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 the structures of related 3-(4-chlorophenylsulfinyl)-2-methyl-1-benzofuran derivatives, see: Choi *et al.* (2010a,b).



Experimental

Crystal data

 $\text{C}_{15}\text{H}_{10}\text{BrClO}_2\text{S}$ $M_r = 369.65$

Monoclinic, $P2_1/c$
 $a = 11.530(6)$ Å
 $b = 5.834(3)$ Å
 $c = 22.045(13)$ Å
 $\beta = 100.602(16)^\circ$
 $V = 1457.6(15)$ Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 3.14$ mm⁻¹
 $T = 173$ K
 $0.20 \times 0.16 \times 0.15$ mm

Data collection

Bruker SMART APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2009)
 $T_{\min} = 0.625$, $T_{\max} = 0.746$

12344 measured reflections
 3169 independent reflections
 2505 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.054$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.125$
 $S = 1.08$
 3169 reflections

182 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.37$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.61$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C9}-\text{H9C}\cdots\text{O2}^i$	0.98	2.51	3.473 (5)	166
$\text{C14}-\text{H14}\cdots\text{O2}^{ii}$	0.95	2.56	3.376 (4)	145

Symmetry codes: (i) $-x + 2, y - \frac{1}{2}, -z + \frac{3}{2}$; (ii) $-x + 2, -y + 2, -z + 1$.

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: HG2719).

References

- Akgul, Y. Y. & Anil, H. (2003). *Phytochemistry*, **63**, 939–943.
 Aslam, S. N., Stevenson, P. C., Phythian, S. J., Veitch, N. C. & Hall, D. R. (2006). *Tetrahedron*, **62**, 4214–4226.
 Brandenburg, K. (1998). DIAMOND. Crystal Impact GbR, Bonn, Germany.
 Bruker (2009). APEX2, SADABS and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
 Choi, H. D., Seo, P. J., Son, B. W. & Lee, U. (2010a). *Acta Cryst.* **E66**, o2325.
 Choi, H. D., Seo, P. J., Son, B. W. & Lee, U. (2010b). *Acta Cryst.* **E66**, o2551.
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Galal, S. A., Abd El-All, A. S., Abdallah, M. M. & El-Diwani, H. I. (2009). *Bioorg. Med. Chem. Lett.* **19**, 2420–2428.
 Khan, M. W., Alam, M. J., Rashid, M. A. & Chowdhury, R. (2005). *Bioorg. Med. Chem.* **13**, 4796–4805.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Soekamto, N. H., Achmad, S. A., Ghisalberty, E. L., Hakim, E. H. & Syah, Y. M. (2003). *Phytochemistry*, **64**, 831–834.

supplementary materials

Acta Cryst. (2010). E66, o2721 [doi:10.1107/S1600536810038870]

5-Bromo-3-(4-chlorophenylsulfinyl)-2-methyl-1-benzofuran

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

Comment

Many compounds involving a benzofuran skeleton have received much attention owing to their important pharmacological properties such as antifungal, antimicrobial, antitumor and antiviral activities (Aslam *et al.*, 2006; Galal *et al.*, 2009; Khan *et al.*, 2005). These compounds widely occur in nature (Akgul & Anil, 2003; Soekamto *et al.*, 2003). As a part of our study of the substituent effect on the solid state structures of 3-(4-chlorophenylsulfinyl)-2-methyl-1-benzofuran analogues (Choi *et al.*, 2010*a,b*), we report herein on the crystal structure of the title compound.

In the title molecule (Fig. 1), the benzofuran unit is essentially planar, with a mean deviation of 0.090 (2) Å from the least-squares plane defined by the nine constituent atoms. The 4-chlorophenyl ring makes a dihedral angle of 89.55 (9)° with the mean plane of the benzofuran fragment. The crystal packing (Fig. 2) is stabilized by weak intermolecular C—H···O hydrogen bonds; the first one between a methyl H atom and the oxygen of the S=O unit [C9—H9C···O2ⁱ; see Table 1], and the second one between the 4-chlorophenyl H atom and the oxygen of the S=O unit [C14—H14···O2ⁱⁱ; see Table 1], respectively. The crystal packing (Fig. 2) is further stabilized by an intermolecular a Br—Brⁱⁱⁱ interaction at 3.783 (3) Å.

Experimental

77% 3-chloroperoxybenzoic acid (202 mg, 0.9 mmol) was added in small portions to a stirred solution of 5-bromo-3-(4-chlorophenylsulfonyl)-2-methyl-1-benzofuran (289 mg, 0.8 mmol) in dichloromethane (30 mL) at 273 K. After being stirred at room temperature for 5h, 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 (silica gel, hexane–ethyl acetate, 1:1 v/v) to afford the title compound as a colorless solid [yield 83%, m.p. 404–405 K; R_f = 0.64 (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 acetone at room temperature.

Refinement

All H atoms were positioned geometrically and refined using riding model, with C—H = 0.93 Å for aryl and 0.98 Å for methyl H atoms. $U_{iso}(H) = 1.2U_{eq}(C)$ for aryl and $1.5U_{eq}(C)$ for methyl H atoms.

Figures

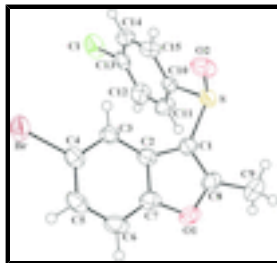


Fig. 1. The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as a small spheres of arbitrary radius.

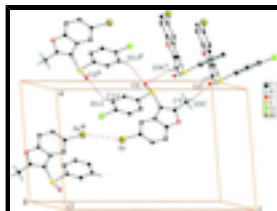


Fig. 2. A view of the C—H...O and Br...Br interactions (dotted lines) in the crystal structure of the title compound. [Symmetry codes: (i) $-x + 2, y - 1/2, -z + 3/2$; (ii) $-x + 2, -y + 2, -z + 1$; (iii) $-x + 1, -y + 3, -z + 1$.]

5-Bromo-3-(4-chlorophenylsulfinyl)-2-methyl-1-benzofuran

Crystal data

$C_{15}H_{10}BrClO_2S$

$M_r = 369.65$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 11.530\ (6)\ \text{\AA}$

$b = 5.834\ (3)\ \text{\AA}$

$c = 22.045\ (13)\ \text{\AA}$

$\beta = 100.602\ (16)^\circ$

$V = 1457.6\ (15)\ \text{\AA}^3$

$Z = 4$

$F(000) = 736$

$D_x = 1.684\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 4598 reflections

$\theta = 2.4\text{--}27.8^\circ$

$\mu = 3.14\ \text{mm}^{-1}$

$T = 173\ \text{K}$

Block, colourless

$0.20 \times 0.16 \times 0.15\ \text{mm}$

Data collection

Bruker SMART APEXII CCD diffractometer

Radiation source: rotating anode graphite multilayer

Detector resolution: $10.0\ \text{pixels mm}^{-1}$

ϕ and ω scans

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

$T_{\min} = 0.625, T_{\max} = 0.746$

12344 measured reflections

3169 independent reflections

2505 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.054$

$\theta_{\max} = 27.0^\circ, \theta_{\min} = 1.8^\circ$

$h = -14 \rightarrow 14$

$k = -7 \rightarrow 7$

$l = -28 \rightarrow 28$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.040$	Hydrogen site location: difference Fourier map
$wR(F^2) = 0.125$	H-atom parameters constrained
$S = 1.08$	$w = 1/[\sigma^2(F_o^2) + (0.0713P)^2]$
3169 reflections	where $P = (F_o^2 + 2F_c^2)/3$
182 parameters	$(\Delta/\sigma)_{\max} = 0.001$
0 restraints	$\Delta\rho_{\max} = 0.37 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -0.61 \text{ e } \text{\AA}^{-3}$

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br	0.49545 (3)	1.26359 (6)	0.558213 (16)	0.05190 (16)
Cl	0.73989 (8)	0.5654 (2)	0.33164 (3)	0.0649 (3)
S	0.93528 (6)	0.61437 (15)	0.61528 (3)	0.0386 (2)
O1	0.69728 (19)	0.4763 (4)	0.71682 (8)	0.0423 (5)
O2	0.9809 (2)	0.8550 (5)	0.62575 (10)	0.0516 (6)
C1	0.8083 (3)	0.5913 (5)	0.64826 (12)	0.0345 (6)
C2	0.7048 (3)	0.7367 (5)	0.64045 (12)	0.0328 (6)
C3	0.6625 (2)	0.9187 (5)	0.60174 (12)	0.0344 (6)
H3	0.7039	0.9731	0.5711	0.041*
C4	0.5576 (3)	1.0161 (5)	0.61011 (12)	0.0371 (6)
C5	0.4941 (3)	0.9380 (6)	0.65489 (13)	0.0433 (7)
H5	0.4221	1.0104	0.6590	0.052*
C6	0.5362 (3)	0.7563 (6)	0.69289 (13)	0.0453 (8)
H6	0.4950	0.7011	0.7235	0.054*
C7	0.6407 (3)	0.6592 (6)	0.68429 (12)	0.0368 (6)
C8	0.7994 (3)	0.4395 (5)	0.69429 (12)	0.0371 (7)
C9	0.8777 (4)	0.2533 (6)	0.72349 (16)	0.0533 (9)
H9A	0.9466	0.2421	0.7035	0.080*
H9B	0.8346	0.1077	0.7189	0.080*

supplementary materials

H9C	0.9037	0.2870	0.7674	0.080*
C10	0.8689 (2)	0.6021 (5)	0.53503 (12)	0.0300 (6)
C11	0.8105 (3)	0.4075 (5)	0.50999 (14)	0.0451 (8)
H11	0.7987	0.2828	0.5359	0.054*
C12	0.7695 (3)	0.3952 (6)	0.44718 (15)	0.0488 (8)
H12	0.7280	0.2639	0.4293	0.059*
C13	0.7902 (2)	0.5784 (6)	0.41089 (13)	0.0390 (7)
C14	0.8486 (3)	0.7708 (5)	0.43509 (15)	0.0434 (8)
H14	0.8616	0.8944	0.4091	0.052*
C15	0.8881 (3)	0.7822 (5)	0.49797 (14)	0.0395 (7)
H15	0.9286	0.9148	0.5157	0.047*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0453 (2)	0.0542 (3)	0.0552 (2)	0.01571 (15)	0.00677 (17)	0.00300 (14)
Cl	0.0517 (5)	0.1073 (8)	0.0349 (4)	0.0106 (5)	0.0053 (4)	-0.0076 (4)
S	0.0285 (4)	0.0529 (5)	0.0344 (4)	0.0028 (3)	0.0059 (3)	0.0022 (3)
O1	0.0496 (13)	0.0516 (13)	0.0274 (9)	-0.0036 (11)	0.0116 (9)	0.0057 (8)
O2	0.0441 (13)	0.0659 (15)	0.0450 (12)	-0.0217 (13)	0.0090 (10)	-0.0112 (11)
C1	0.0330 (15)	0.0442 (17)	0.0269 (12)	0.0012 (13)	0.0067 (11)	0.0009 (11)
C2	0.0307 (15)	0.0414 (17)	0.0263 (12)	-0.0052 (12)	0.0054 (11)	-0.0035 (10)
C3	0.0310 (14)	0.0427 (17)	0.0312 (13)	-0.0006 (13)	0.0099 (11)	0.0014 (11)
C4	0.0320 (14)	0.0464 (17)	0.0323 (13)	0.0004 (14)	0.0040 (12)	-0.0075 (12)
C5	0.0300 (15)	0.064 (2)	0.0374 (14)	0.0020 (15)	0.0105 (12)	-0.0127 (14)
C6	0.0412 (18)	0.068 (2)	0.0301 (15)	-0.0078 (15)	0.0159 (14)	-0.0050 (13)
C7	0.0394 (16)	0.0462 (17)	0.0252 (12)	-0.0063 (14)	0.0067 (12)	-0.0033 (12)
C8	0.0436 (16)	0.0414 (16)	0.0257 (12)	-0.0029 (14)	0.0046 (12)	-0.0003 (11)
C9	0.068 (3)	0.051 (2)	0.0385 (16)	0.0065 (16)	0.0032 (17)	0.0119 (13)
C10	0.0256 (13)	0.0325 (14)	0.0334 (13)	0.0047 (11)	0.0096 (11)	0.0010 (10)
C11	0.054 (2)	0.0348 (16)	0.0470 (16)	-0.0054 (15)	0.0095 (15)	0.0040 (13)
C12	0.052 (2)	0.0419 (18)	0.0496 (17)	-0.0068 (16)	0.0025 (15)	-0.0083 (14)
C13	0.0278 (14)	0.058 (2)	0.0336 (14)	0.0102 (14)	0.0110 (12)	-0.0027 (13)
C14	0.0409 (18)	0.052 (2)	0.0395 (15)	0.0005 (15)	0.0137 (14)	0.0109 (13)
C15	0.0374 (17)	0.0410 (17)	0.0424 (15)	-0.0088 (13)	0.0132 (14)	-0.0017 (12)

Geometric parameters (\AA , $^\circ$)

Br—C4	1.899 (3)	C6—C7	1.376 (5)
Br—Br ⁱ	3.7826 (17)	C6—H6	0.9500
Cl—C13	1.738 (3)	C8—C9	1.482 (4)
S—O2	1.502 (3)	C9—H9A	0.9800
S—C1	1.756 (3)	C9—H9B	0.9800
S—C10	1.795 (3)	C9—H9C	0.9800
O1—C8	1.376 (4)	C10—C15	1.373 (4)
O1—C7	1.381 (4)	C10—C11	1.382 (4)
C1—C8	1.365 (4)	C11—C12	1.380 (4)
C1—C2	1.448 (4)	C11—H11	0.9500

C2—C3	1.393 (4)	C12—C13	1.382 (5)
C2—C7	1.395 (4)	C12—H12	0.9500
C3—C4	1.379 (4)	C13—C14	1.366 (5)
C3—H3	0.9500	C14—C15	1.379 (4)
C4—C5	1.409 (4)	C14—H14	0.9500
C5—C6	1.383 (5)	C15—H15	0.9500
C5—H5	0.9500		
C4—Br—Br ⁱ	155.35 (9)	C1—C8—C9	132.5 (3)
O2—S—C1	107.65 (14)	O1—C8—C9	116.8 (3)
O2—S—C10	105.22 (13)	C8—C9—H9A	109.5
C1—S—C10	99.69 (13)	C8—C9—H9B	109.5
C8—O1—C7	106.7 (2)	H9A—C9—H9B	109.5
C8—C1—C2	107.2 (3)	C8—C9—H9C	109.5
C8—C1—S	122.7 (2)	H9A—C9—H9C	109.5
C2—C1—S	129.6 (2)	H9B—C9—H9C	109.5
C3—C2—C7	120.1 (3)	C15—C10—C11	120.7 (3)
C3—C2—C1	135.0 (3)	C15—C10—S	118.0 (2)
C7—C2—C1	104.9 (2)	C11—C10—S	120.9 (2)
C4—C3—C2	116.6 (3)	C12—C11—C10	119.7 (3)
C4—C3—H3	121.7	C12—C11—H11	120.2
C2—C3—H3	121.7	C10—C11—H11	120.2
C3—C4—C5	122.9 (3)	C11—C12—C13	118.5 (3)
C3—C4—Br	118.4 (2)	C11—C12—H12	120.8
C5—C4—Br	118.7 (2)	C13—C12—H12	120.8
C6—C5—C4	120.1 (3)	C14—C13—C12	122.3 (3)
C6—C5—H5	119.9	C14—C13—Cl	118.5 (2)
C4—C5—H5	119.9	C12—C13—Cl	119.1 (2)
C7—C6—C5	116.8 (3)	C13—C14—C15	118.7 (3)
C7—C6—H6	121.6	C13—C14—H14	120.7
C5—C6—H6	121.6	C15—C14—H14	120.7
C6—C7—O1	126.1 (3)	C10—C15—C14	120.1 (3)
C6—C7—C2	123.4 (3)	C10—C15—H15	120.0
O1—C7—C2	110.5 (3)	C14—C15—H15	120.0
C1—C8—O1	110.7 (3)		
O2—S—C1—C8	-120.0 (3)	C1—C2—C7—O1	-1.0 (3)
C10—S—C1—C8	130.5 (2)	C2—C1—C8—O1	0.0 (3)
O2—S—C1—C2	50.7 (3)	S—C1—C8—O1	172.54 (19)
C10—S—C1—C2	-58.8 (3)	C2—C1—C8—C9	-178.9 (3)
C8—C1—C2—C3	-179.5 (3)	S—C1—C8—C9	-6.4 (5)
S—C1—C2—C3	8.7 (5)	C7—O1—C8—C1	-0.7 (3)
C8—C1—C2—C7	0.6 (3)	C7—O1—C8—C9	178.4 (3)
S—C1—C2—C7	-171.2 (2)	O2—S—C10—C15	10.8 (3)
C7—C2—C3—C4	1.0 (4)	C1—S—C10—C15	122.2 (2)
C1—C2—C3—C4	-178.9 (3)	O2—S—C10—C11	-175.8 (2)
C2—C3—C4—C5	-0.4 (4)	C1—S—C10—C11	-64.3 (3)
C2—C3—C4—Br	-179.6 (2)	C15—C10—C11—C12	-0.9 (5)
C3—C4—C5—C6	0.0 (5)	S—C10—C11—C12	-174.2 (3)
Br—C4—C5—C6	179.2 (2)	C10—C11—C12—C13	1.1 (5)

supplementary materials

C4—C5—C6—C7	-0.2 (4)	C11—C12—C13—C14	-0.7 (5)
C5—C6—C7—O1	-179.5 (3)	C11—C12—C13—Cl	179.7 (3)
C5—C6—C7—C2	0.8 (5)	C12—C13—C14—C15	0.0 (5)
C8—O1—C7—C6	-178.6 (3)	Cl—C13—C14—C15	179.7 (3)
C8—O1—C7—C2	1.1 (3)	C11—C10—C15—C14	0.3 (5)
C3—C2—C7—C6	-1.3 (4)	S—C10—C15—C14	173.8 (3)
C1—C2—C7—C6	178.7 (3)	C13—C14—C15—C10	0.2 (5)
C3—C2—C7—O1	179.0 (2)		

Symmetry codes: (i) $-x+1, -y+3, -z+1$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C9—H9C \cdots O2 ⁱⁱ	0.98	2.51	3.473 (5)	166
C14—H14 \cdots O2 ⁱⁱⁱ	0.95	2.56	3.376 (4)	145

Symmetry codes: (ii) $-x+2, y-1/2, -z+3/2$; (iii) $-x+2, -y+2, -z+1$.

Fig. 1

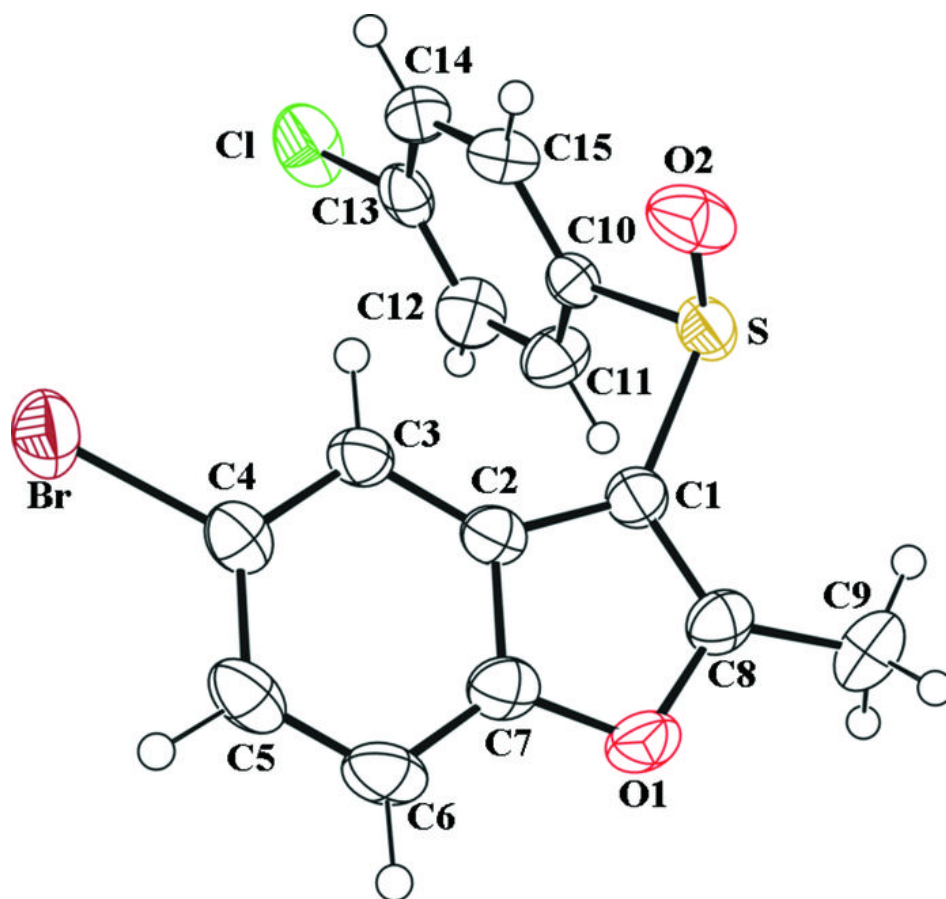


Fig. 2

