

2-Chloroethyl 2-(5-bromo-3-methylsulfinyl-1-benzofuran-2-yl)acetate

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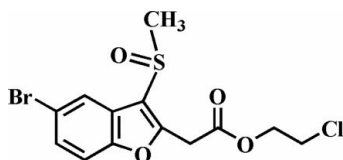
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.008$ Å; disorder in main residue; R factor = 0.055; wR factor = 0.133; data-to-parameter ratio = 13.4.

In the title compound, $\text{C}_{13}\text{H}_{12}\text{BrClO}_4\text{S}$, the O atom and the methyl group of the methylsulfinyl substituent lie on opposite sides of the plane of the benzofuran fragment. There is a mean deviation of 0.016 (4) Å from the least-squares plane defined by the nine constituent benzofuran atoms. The crystal structure is stabilized by aromatic $\pi-\pi$ interactions between the benzene rings of neighbouring molecules [centroid-centroid distance = 3.689 (7) Å] and by a weak $\text{C}-\text{H}\cdots\pi$ interaction between an H atom of the methylene group bonded to the carboxylate O atom and the benzene ring of an adjacent molecule. In addition, the crystal structure exhibits weak non-classical intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds. The chloroethyl group is disordered over two positions, with refined site-occupancy factors of 0.767 (6) and 0.233 (6).

Related literature

For the crystal structures of similar alkyl 2-(5-bromo-3-methylsulfinyl-1-benzofuran-2-yl)acetate derivatives, see: Choi *et al.* (2008a,b).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{12}\text{BrClO}_4\text{S}$
 $M_r = 379.65$
 Triclinic, $P\bar{1}$
 $a = 8.495$ (1) Å
 $b = 9.882$ (2) Å
 $c = 10.277$ (2) Å
 $\alpha = 71.095$ (3)°
 $\beta = 80.331$ (3)°
 $\gamma = 65.012$ (2)°
 $V = 739.3$ (2) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 3.11$ mm⁻¹
 $T = 298$ K
 $0.30 \times 0.20 \times 0.10$ mm

Data collection

Bruker SMART CCD diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1999)
 $T_{\min} = 0.475$, $T_{\max} = 0.736$
 5479 measured reflections
 2575 independent reflections
 1972 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.055$
 $wR(F^2) = 0.133$
 $S = 1.07$
 2575 reflections
 192 parameters
 57 restraints
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.80$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.81$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C11A}-\text{H11B}\cdots\text{Cg}^i$	0.97	3.07	3.779 (7)	131
$\text{C3}-\text{H3}\cdots\text{O4}^{ii}$	0.93	2.60	3.483 (7)	159
$\text{C5}-\text{H5}\cdots\text{O3}^{iii}$	0.93	2.58	3.423 (7)	150
$\text{C9}-\text{H9A}\cdots\text{O1}^{iv}$	0.97	2.60	3.545 (6)	165
$\text{C9}-\text{H9B}\cdots\text{O4}^v$	0.97	2.34	3.294 (7)	170

Symmetry codes: (i) $x, y - 1, z$; (ii) $-x + 1, -y + 1, -z + 1$; (iii) $-x + 1, -y + 1, -z$; (iv) $-x, -y + 1, -z$; (v) $-x, -y + 1, -z + 1$. 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, 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: SJ2602).

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Acta Cryst. (2009). E65, o1025 [doi:10.1107/S1600536809012847]

2-Chloroethyl 2-(5-bromo-3-methylsulfinyl-1-benzofuran-2-yl)acetate

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Comment

This work is related to our previous communications on the synthesis and structure of alkyl 2-(5-bromo-3-methylsulfinyl-1-benzofuran-2-yl)acetate analogues, viz. isopropyl 2-(5-bromo-3-methylsulfinyl-1-benzofuran-2-yl)acetate (Choi *et al.*, 2008a) and methyl 2-(5-bromo-3-methylsulfinyl-1-benzofuran-2-yl)acetate (Choi *et al.*, 2008b). Here we report the crystal structure of the title compound, 2-chloroethyl 2-(5-bromo-3-methylsulfinyl-1-benzofuran-2-yl)acetate (Fig. 1).

The benzofuran unit is essentially planar, with a mean deviation of 0.016 (4) Å from the least-squares plane defined by the nine constituent atoms. The chloroethyl group is disordered over two positions with site-occupancy factors of 0.767 (6) (for atoms labelled A) and 0.233 (7) (for atoms labelled B). The molecular packing (Fig. 2) is stabilized by aromatic π - π interactions between the benzene rings of neighbouring molecules, with a $\text{Cg}\cdots\text{Cg}^{\text{iii}}$ distance of 3.689 (7) Å (Cg is the centroid of the C2-C7 benzene ring; symmetry codes as in Fig. 2). The crystal packing is further stabilized by an intermolecular $\text{C}-\text{H}\cdots\pi$ interaction between an H atom of the methylene group bonded to the carboxylate O atom and the benzene ring of a neighbouring molecule, with a $\text{C11}-\text{H11B}\cdots\text{Cg}^{\text{i}}$ distance of 3.07 Å (Table 1 and Fig. 2; symmetry code as in Fig. 2, Cg^{i} is the centroid of the C2-C7 benzene ring). Additionally, the crystal structure exhibits weak, non-classical, intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds; the first between a benzene H atom and the $\text{S}=\text{O}$ unit, the second between a benzene H atom and the $\text{C}=\text{O}$ unit, the third between an H atom of the methylene group bonded to the carboxylate C atom and the furan O atom, and the fourth between an H atom of the methylene group bonded to the carboxylate C atom and the $\text{S}=\text{O}$ unit, respectively (Table 1 and Fig. 3; symmetry codes as in Fig. 3).

Experimental

77% 3-Chloroperoxybenzoic acid (271 mg, 1.1 mmol) was added in small portions to a stirred solution of 2-chloroethyl 2-(5-bromo-3-methylsulfonyl-1-benzofuran-2-yl)acetate (380 mg, 1.0 mmol) in dichloromethane (40 ml) at 273 K. After stirring for 3 h at room temperature, 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, 1:2 v/v) to afford the title compound as a colorless solid [yield 80%, m.p. 436-437 K; R_f = 0.41 (hexane-ethyl acetate, 1:2 v/v)]. Single crystals suitable for X-ray diffraction were prepared by evaporation of a solution of the title compound in chloroform at room temperature. Spectroscopic analysis: ^1H NMR (CDCl_3 , 400 MHz) δ 3.07 (s, 3H), 3.70 (t, J = 5.48 Hz, 2H), 4.12 (s, 2H), 4.41 (t, J = 5.48 Hz, 2H), 7.41 (d, J = 8.76 Hz, 1H), 7.71 (dd, J = 8.47 Hz and J = 1.82 Hz, 1H), 8.05 (s, 1H).

Refinement

All H atoms were positioned geometrically and refined using a riding model, with $\text{C}-\text{H}$ = 0.93 Å for the aryl, 0.97 Å for the methylene, and 0.96 Å for the methyl H atoms. $\text{Uiso}(\text{H}) = 1.2\text{Ueq}(\text{C})$ for the aryl and methylene H atoms, and $1.5\text{Ueq}(\text{C})$ for methyl H atoms. The chloroethyl group is disordered over two positions, with site-occupancy factors, from refinement,

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of 0.767 (6) (C11A—C12A—C1A) and 0.233 (7) (C11B—C12B—C1B). Both sets of C and Cl atoms were restrained using the commands ISOR (0.01), EADP, and the C—C and C—Cl distances (A & B) were restrained to 1.46 (3) and 1.55 (3) Å, respectively, using the command DFIX. Despite this the atomic displacement parameters for the C atoms of the disordered chloroethyl group were large, reflecting additional disorder and the room temperature data collection.

Figures

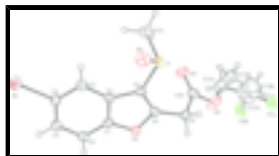


Fig. 1. The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as a small spheres of arbitrary radius.

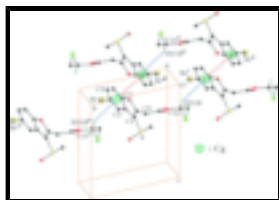


Fig. 2. The π – π and C—H \cdots π interactions (dotted lines) in the title compound. Cg denotes the ring centroid of the C2 \cdots C7 benzene ring. The disordered component of the chloroethyl group, part B, has been omitted for clarity as have H atoms not involved in intermolecular contacts. [Symmetry codes: (i) $x, y-1, z$; (ii) $-x+1, -y, -z$; (iii) $-x+1, -y+1, -z$; (iv) $x, y+1, z$.]

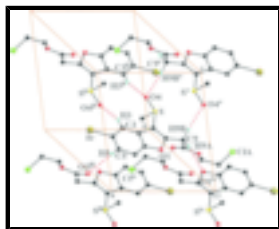


Fig. 3. The C—H \cdots O hydrogen bonds (dotted lines) in the title compound. The disordered component of the chloroethyl group, part B, has been omitted for clarity as have H atoms not involved in intermolecular contacts. [Symmetry codes: (ii) $-x+1, -y+1, -z+1$; (iii) $-x+1, -y+1, -z$; (iv) $-x, -y+1, -z$ (v) $-x, -y+1, -z+1$.]

2-Chloroethyl 2-(5-bromo-3-methylsulfinyl-1-benzofuran-2-yl)acetate

Crystal data

$C_{13}H_{12}BrClO_4S$	$Z = 2$
$M_r = 379.65$	$F_{000} = 380$
Triclinic, $P\bar{1}$	$D_x = 1.705 \text{ Mg m}^{-3}$
Hall symbol: $-p 1$	Mo $K\alpha$ radiation
$a = 8.495 (1) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 9.882 (2) \text{ \AA}$	Cell parameters from 2253 reflections
$c = 10.277 (2) \text{ \AA}$	$\theta = 2.7\text{--}25.7^\circ$
$\alpha = 71.095 (3)^\circ$	$\mu = 3.11 \text{ mm}^{-1}$
$\beta = 80.331 (3)^\circ$	$T = 298 \text{ K}$
$\gamma = 65.012 (2)^\circ$	Block, colorless
$V = 739.3 (2) \text{ \AA}^3$	$0.30 \times 0.20 \times 0.10 \text{ mm}$

Data collection

Bruker SMART CCD diffractometer	2575 independent reflections
Radiation source: fine-focus sealed tube	1972 reflections with $I > 2\sigma(I)$

Monochromator: graphite $R_{\text{int}} = 0.027$
 Detector resolution: 10.0 pixels mm^{-1} $\theta_{\text{max}} = 25.0^\circ$
 $T = 298 \text{ K}$ $\theta_{\text{min}} = 2.1^\circ$
 φ and ω scans $h = -10 \rightarrow 10$
 Absorption correction: multi-scan (SADABS; Sheldrick, 1999) $k = -11 \rightarrow 11$
 $T_{\text{min}} = 0.475$, $T_{\text{max}} = 0.736$ $l = -12 \rightarrow 12$
 5479 measured reflections

Refinement

Refinement on F^2 Secondary atom site location: difference Fourier map
 Least-squares matrix: full Hydrogen site location: difference Fourier map
 $R[F^2 > 2\sigma(F^2)] = 0.055$ H-atom parameters constrained
 $wR(F^2) = 0.133$ $w = 1/[\sigma^2(F_o^2) + (0.054P)^2 + 1.6319P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.07$ $(\Delta/\sigma)_{\text{max}} < 0.000$
 2575 reflections $\Delta\rho_{\text{max}} = 0.80 \text{ e } \text{\AA}^{-3}$
 192 parameters $\Delta\rho_{\text{min}} = -0.80 \text{ e } \text{\AA}^{-3}$
 57 restraints Extinction correction: none
 Primary atom site location: structure-invariant direct methods

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>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Br	0.68390 (9)	0.78885 (8)	0.10766 (7)	0.0571 (3)	
ClA	-0.1810 (4)	-0.0480 (3)	0.3580 (3)	0.0928 (12)	0.764 (6)
ClB	-0.0308 (13)	-0.0729 (11)	0.1579 (12)	0.0928 (12)	0.24
S	0.25778 (18)	0.40156 (16)	0.46589 (13)	0.0391 (4)	
O1	0.1648 (4)	0.5435 (4)	0.0705 (3)	0.0355 (8)	
O2	0.0404 (6)	0.1375 (5)	0.2446 (7)	0.0824 (18)	
O3	0.2843 (6)	0.1340 (5)	0.2991 (5)	0.0622 (12)	
O4	0.2572 (6)	0.5265 (5)	0.5185 (4)	0.0537 (11)	
C1	0.2533 (6)	0.4767 (6)	0.2849 (5)	0.0313 (11)	

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C2	0.3429 (6)	0.5699 (5)	0.1946 (5)	0.0287 (11)	
C3	0.4664 (7)	0.6219 (6)	0.2093 (6)	0.0362 (12)	
H3	0.5126	0.5960	0.2941	0.043*	
C4	0.5167 (7)	0.7138 (6)	0.0920 (6)	0.0379 (13)	
C5	0.4497 (7)	0.7563 (6)	-0.0359 (6)	0.0395 (13)	
H5	0.4849	0.8216	-0.1110	0.047*	
C6	0.3306 (7)	0.7020 (6)	-0.0522 (6)	0.0382 (13)	
H6	0.2860	0.7271	-0.1375	0.046*	
C7	0.2815 (6)	0.6090 (6)	0.0641 (5)	0.0326 (12)	
C8	0.1505 (7)	0.4640 (6)	0.2065 (5)	0.0328 (11)	
C9	0.0354 (7)	0.3767 (6)	0.2371 (6)	0.0377 (13)	
H9A	-0.0383	0.4141	0.1598	0.045*	
H9B	-0.0392	0.3968	0.3170	0.045*	
C10	0.1380 (8)	0.2042 (7)	0.2639 (6)	0.0446 (14)	
C11A	0.1269 (13)	-0.0271 (11)	0.2556 (12)	0.069 (3)	0.764 (6)
H11A	0.2031	-0.0798	0.3329	0.083*	0.764 (6)
H11B	0.1968	-0.0422	0.1725	0.083*	0.764 (6)
C12A	-0.004 (3)	-0.092 (3)	0.275 (2)	0.162 (6)	0.764 (6)
H12A	0.0570	-0.2031	0.3143	0.195*	0.764 (6)
H12B	-0.0332	-0.0779	0.1832	0.195*	0.764 (6)
C11B	0.107 (4)	-0.044 (3)	0.343 (4)	0.069 (3)	0.24
H11C	0.2242	-0.1045	0.3153	0.083*	0.236 (6)
H11D	0.1055	-0.0480	0.4385	0.083*	0.236 (6)
C12B	-0.013 (10)	-0.105 (10)	0.324 (4)	0.162 (6)	0.24
H12C	-0.1259	-0.0542	0.3648	0.195*	0.236 (6)
H12D	0.0286	-0.2156	0.3696	0.195*	0.236 (6)
C13	0.4757 (8)	0.2580 (7)	0.4793 (7)	0.0566 (17)	
H13A	0.5553	0.3084	0.4440	0.085*	
H13B	0.4904	0.1885	0.4269	0.085*	
H13C	0.4983	0.2002	0.5740	0.085*	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0555 (4)	0.0544 (4)	0.0722 (5)	-0.0345 (3)	-0.0077 (3)	-0.0112 (3)
ClA	0.086 (2)	0.0646 (16)	0.118 (2)	-0.0415 (14)	0.0202 (16)	-0.0107 (14)
ClB	0.086 (2)	0.0646 (16)	0.118 (2)	-0.0415 (14)	0.0202 (16)	-0.0107 (14)
S	0.0433 (8)	0.0443 (8)	0.0292 (7)	-0.0198 (7)	-0.0030 (6)	-0.0056 (6)
O1	0.039 (2)	0.041 (2)	0.0288 (19)	-0.0193 (17)	-0.0074 (16)	-0.0062 (16)
O2	0.044 (3)	0.040 (3)	0.174 (6)	-0.012 (2)	-0.022 (3)	-0.040 (3)
O3	0.045 (3)	0.046 (3)	0.087 (3)	-0.014 (2)	-0.021 (2)	-0.006 (2)
O4	0.061 (3)	0.065 (3)	0.041 (2)	-0.022 (2)	-0.001 (2)	-0.027 (2)
C1	0.033 (3)	0.033 (3)	0.030 (3)	-0.013 (2)	-0.005 (2)	-0.009 (2)
C2	0.030 (3)	0.023 (2)	0.029 (3)	-0.005 (2)	-0.002 (2)	-0.009 (2)
C3	0.039 (3)	0.033 (3)	0.038 (3)	-0.012 (2)	-0.004 (2)	-0.014 (2)
C4	0.039 (3)	0.033 (3)	0.049 (3)	-0.017 (2)	-0.002 (3)	-0.015 (3)
C5	0.040 (3)	0.035 (3)	0.040 (3)	-0.015 (2)	0.001 (3)	-0.007 (2)
C6	0.042 (3)	0.039 (3)	0.030 (3)	-0.014 (3)	-0.004 (2)	-0.007 (2)

C7	0.029 (3)	0.028 (3)	0.039 (3)	-0.007 (2)	-0.006 (2)	-0.012 (2)
C8	0.033 (3)	0.031 (3)	0.033 (3)	-0.012 (2)	-0.001 (2)	-0.009 (2)
C9	0.036 (3)	0.045 (3)	0.037 (3)	-0.020 (3)	-0.005 (2)	-0.012 (3)
C10	0.045 (4)	0.041 (3)	0.048 (4)	-0.016 (3)	-0.003 (3)	-0.012 (3)
C11A	0.059 (4)	0.062 (4)	0.090 (5)	-0.019 (3)	-0.002 (4)	-0.032 (4)
C12A	0.170 (8)	0.120 (7)	0.171 (9)	-0.051 (6)	0.035 (7)	-0.037 (7)
C11B	0.059 (4)	0.062 (4)	0.090 (5)	-0.019 (3)	-0.002 (4)	-0.032 (4)
C12B	0.170 (8)	0.120 (7)	0.171 (9)	-0.051 (6)	0.035 (7)	-0.037 (7)
C13	0.055 (4)	0.051 (4)	0.057 (4)	-0.010 (3)	-0.017 (3)	-0.012 (3)

Geometric parameters (Å, °)

Br—C4	1.907 (5)	C5—H5	0.9300
C1A—C12A	1.559 (19)	C6—C7	1.375 (7)
C1B—C12B	1.65 (3)	C6—H6	0.9300
S—O4	1.498 (4)	C8—C9	1.494 (7)
S—C1	1.766 (5)	C9—C10	1.505 (8)
S—C13	1.788 (6)	C9—H9A	0.9700
O1—C8	1.377 (6)	C9—H9B	0.9700
O1—C7	1.378 (6)	C11A—C12A	1.459 (15)
O2—C10	1.328 (7)	C11A—H11A	0.9700
O2—C11A	1.448 (9)	C11A—H11B	0.9700
O2—C11B	1.65 (2)	C12A—H12A	0.9700
O3—C10	1.190 (7)	C12A—H12B	0.9700
C1—C8	1.350 (7)	C11B—C12B	1.45 (2)
C1—C2	1.445 (7)	C11B—H11C	0.9700
C2—C3	1.395 (7)	C11B—H11D	0.9700
C2—C7	1.396 (7)	C12B—H12C	0.9700
C3—C4	1.380 (7)	C12B—H12D	0.9700
C3—H3	0.9300	C13—H13A	0.9600
C4—C5	1.385 (8)	C13—H13B	0.9600
C5—C6	1.383 (7)	C13—H13C	0.9600
O4—S—C1	105.9 (2)	C10—C9—H9B	109.2
O4—S—C13	106.1 (3)	H9A—C9—H9B	107.9
C1—S—C13	98.5 (3)	O3—C10—O2	123.5 (5)
C8—O1—C7	106.5 (4)	O3—C10—C9	126.4 (5)
C10—O2—C11A	116.1 (6)	O2—C10—C9	110.0 (5)
C10—O2—C11B	109.4 (13)	O2—C11A—C12A	108.9 (13)
C11A—O2—C11B	31.3 (12)	O2—C11A—H11A	109.9
C8—C1—C2	107.4 (4)	C12A—C11A—H11A	109.9
C8—C1—S	123.8 (4)	O2—C11A—H11B	109.9
C2—C1—S	128.7 (4)	C12A—C11A—H11B	109.9
C3—C2—C7	119.1 (5)	H11A—C11A—H11B	108.3
C3—C2—C1	136.0 (5)	C11A—C12A—C1A	128.4 (16)
C7—C2—C1	104.9 (4)	C11A—C12A—H12A	105.2
C4—C3—C2	116.8 (5)	C1A—C12A—H12A	105.2
C4—C3—H3	121.6	C11A—C12A—H12B	105.2
C2—C3—H3	121.6	C1A—C12A—H12B	105.2
C3—C4—C5	123.3 (5)	H12A—C12A—H12B	105.9

supplementary materials

C3—C4—Br	118.2 (4)	C12B—C11B—O2	106 (4)
C5—C4—Br	118.4 (4)	C12B—C11B—H11C	110.5
C6—C5—C4	120.3 (5)	O2—C11B—H11C	110.5
C6—C5—H5	119.9	C12B—C11B—H11D	110.5
C4—C5—H5	119.9	O2—C11B—H11D	110.5
C7—C6—C5	116.6 (5)	H11C—C11B—H11D	108.7
C7—C6—H6	121.7	C11B—C12B—C1B	110 (3)
C5—C6—H6	121.7	C11B—C12B—H12C	109.7
C6—C7—O1	126.0 (5)	C1B—C12B—H12C	109.7
C6—C7—C2	123.8 (5)	C11B—C12B—H12D	109.7
O1—C7—C2	110.3 (4)	C1B—C12B—H12D	109.7
C1—C8—O1	110.9 (4)	H12C—C12B—H12D	108.2
C1—C8—C9	133.1 (5)	S—C13—H13A	109.5
O1—C8—C9	115.9 (4)	S—C13—H13B	109.5
C8—C9—C10	112.0 (4)	H13A—C13—H13B	109.5
C8—C9—H9A	109.2	S—C13—H13C	109.5
C10—C9—H9A	109.2	H13A—C13—H13C	109.5
C8—C9—H9B	109.2	H13B—C13—H13C	109.5
O4—S—C1—C8	-135.7 (5)	C1—C2—C7—O1	-1.1 (5)
C13—S—C1—C8	114.8 (5)	C2—C1—C8—O1	-0.5 (6)
O4—S—C1—C2	39.3 (5)	S—C1—C8—O1	175.4 (3)
C13—S—C1—C2	-70.2 (5)	C2—C1—C8—C9	176.0 (5)
C8—C1—C2—C3	-178.3 (6)	S—C1—C8—C9	-8.1 (9)
S—C1—C2—C3	6.0 (9)	C7—O1—C8—C1	-0.1 (5)
C8—C1—C2—C7	0.9 (5)	C7—O1—C8—C9	-177.3 (4)
S—C1—C2—C7	-174.7 (4)	C1—C8—C9—C10	-71.8 (8)
C7—C2—C3—C4	1.7 (7)	O1—C8—C9—C10	104.6 (5)
C1—C2—C3—C4	-179.1 (5)	C11A—O2—C10—O3	-5.9 (11)
C2—C3—C4—C5	0.7 (8)	C11B—O2—C10—O3	27.3 (16)
C2—C3—C4—Br	179.5 (4)	C11A—O2—C10—C9	175.0 (7)
C3—C4—C5—C6	-2.4 (8)	C11B—O2—C10—C9	-151.7 (14)
Br—C4—C5—C6	178.8 (4)	C8—C9—C10—O3	21.9 (9)
C4—C5—C6—C7	1.5 (8)	C8—C9—C10—O2	-159.1 (5)
C5—C6—C7—O1	179.8 (5)	C10—O2—C11A—C12A	162.3 (11)
C5—C6—C7—C2	0.9 (8)	C11B—O2—C11A—C12A	78 (3)
C8—O1—C7—C6	-178.2 (5)	O2—C11A—C12A—C1A	-36 (3)
C8—O1—C7—C2	0.8 (5)	C10—O2—C11B—C12B	176 (3)
C3—C2—C7—C6	-2.6 (8)	C11A—O2—C11B—C12B	-76 (4)
C1—C2—C7—C6	178.0 (5)	O2—C11B—C12B—C1B	52 (6)
C3—C2—C7—O1	178.4 (4)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C11A—H11B \cdots Cg ⁱ	0.97	3.07	3.779 (7)	131
C3—H3 \cdots O4 ⁱⁱ	0.93	2.60	3.483 (7)	159
C5—H5 \cdots O3 ⁱⁱⁱ	0.93	2.58	3.423 (7)	150
C9—H9A \cdots O1 ^{iv}	0.97	2.60	3.545 (6)	165

C9—H9B...O4^v 0.97 2.34 3.294 (7) 170
 Symmetry codes: (i) $x, y-1, z$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+1, -y+1, -z$; (iv) $-x, -y+1, -z$; (v) $-x, -y+1, -z+1$.

Fig. 1

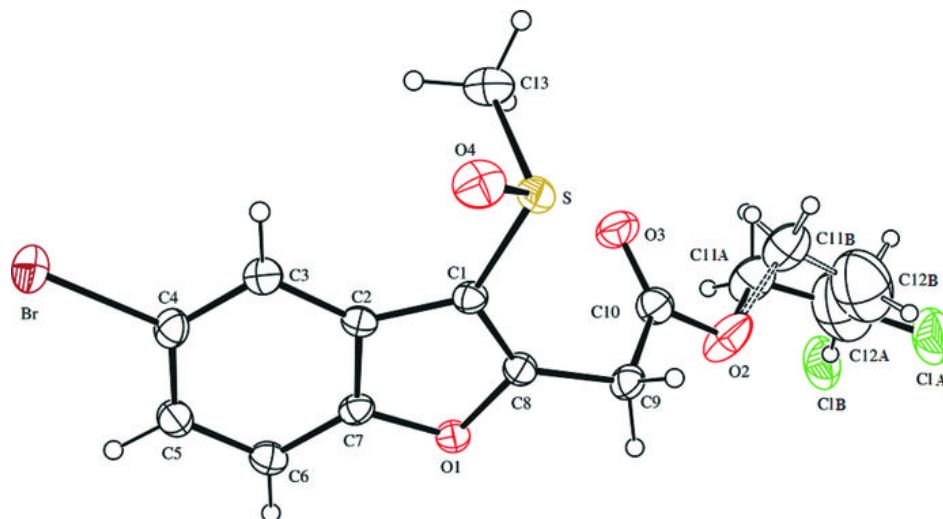


Fig. 2

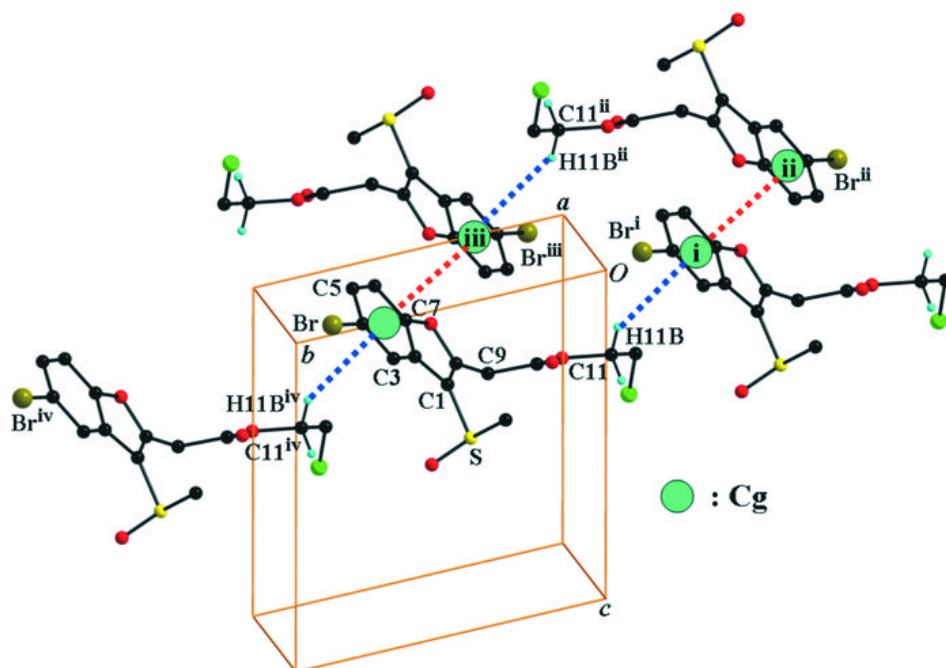


Fig. 3

