Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Ethyl 2-(5-iodo-3-methylsulfinyl-1-benzofuran-2-yl)acetate

Hong Dae Choi,[a] Pil Ja Seo,[a] Byeng Wha Son[b] and Uk Lee[b]*

[a]Department of Chemistry, Dongeui University, San 24 Kaya-dong Busanjin-gu, Busan 614-714, Republic of Korea, and [b]Department 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

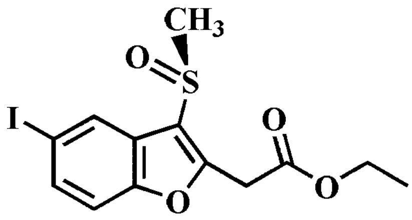
Received 7 September 2007; accepted 12 September 2007

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.004 Å; R factor = 0.020; wR factor = 0.055; data-to-parameter ratio = 17.8.

The title compound, $C_{13}H_{13}IO_4S$, was prepared by the oxidation of ethyl 2-(5-iodo-3-methylsulfanyl-1-benzofuran-2-yl)acetate 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 system. The crystal structure is stabilized by intermolecular aromatic π–π interactions, with a centroid–centroid distance of 3.591 (3) Å between the benzene rings of neighboring molecules, and by three intermolecular C—H$\cdots$O hydrogen bonds.

Related literature

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



Experimental

Crystal data

$C_{13}H_{13}IO_4S$
M_r = 392.19
Triclinic, $P\bar{1}$
a = 8.4311 (4) Å
b = 9.8128 (5) Å
c = 10.0299 (5) Å
α = 70.629 (1)°
β = 78.623 (1)°
γ = 65.370 (1)°
V = 709.92 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.41 mm^{-1}
T = 173 (2) K
0.30 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1999)
$T_{\min}$ = 0.561, $T_{\max}$ = 0.790
6175 measured reflections
3053 independent reflections
2900 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.055
S = 1.08
3053 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 1.08 e Å^{-3}
$\Delta\rho_{\min}$ = −0.54 e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C9—H9$B\cdots$O1$^{\rm i}$	0.99	2.53	3.513 (3)	174
C9—H9$A\cdots$O2$^{\rm ii}$	0.99	2.20	3.190 (3)	174
C3—H3$\cdots$O2$^{\rm iii}$	0.95	2.48	3.391 (3)	161

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

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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2138).

References

Brandenburg, K. (1998). *DIAMOND*. Version 2.1. Crystal Impact GbR, Bonn, Germany.
Bruker (1997). *SMART* (Version 5.631) and *SAINT* (Version 6.12). Bruker AXS Inc., Madison, Wisconsin, USA.
Choi, H. D., Seo, P. J., Son, B. W. & Lee, U. (2007*a*). *Acta Cryst.* E**63**, o3832.
Choi, H. D., Seo, P. J., Son, B. W. & Lee, U. (2007*b*). *Acta Cryst.* E**63**, o3850.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1999). *SADABS*. University of Göttingen, Germany.

doi:10.1107/S1600536807044583

supplementary materials

Acta Cryst. (2007). E**63**, o4081 [**doi:10.1107/S1600536807044583**]

Ethyl 2-(5-iodo-3-methylsulfinyl-1-benzofuran-2-yl)acetate

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

Comment

As part of our continuing studies on the synthesis and structure of ethyl 2-benzofuranacetate analogues, we have recently described the crystal structures of ethyl 2-(5-chloro-3-methylsulfinyl-1-benzofuran-2-yl)acetate (Choi *et al.*, 2007*a*) and ethyl 2-(5-bromo-3-methylsulfinyl-1-benzofuran-2-yl)acetate (Choi *et al.*, 2007*b*). Here we report the crystal structure of the title compound, ethyl 2-(5-iodo-3-methylsulfinyl-1-benzofuran-2-yl)acetate (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 molecular packing (Fig. 2) is stabilized by $\pi\cdots\pi$ stacking interactions between adjacent benzene units. The $Cg\cdots Cg^{i}$ distance is 3.591 (3) Å (Cg is the centroid of the C2—C7 benzene ring; symmetry code as in Fig. 2). The molecular packing is further stabilized by C—H···O hydrogen bonds (Table 1 and Fig. 2); one between a benzene-H and the S═O unit, *i.e.* $C3—H3\cdots O2^{iv}$, a second between a methylene—H and the S═O unit, *i.e.* $C9—H9A\cdots O2^{iii}$, and a third between the second methylene—H and the furan—O, *i.e.* $C9—H9B\cdots O1^{ii}$, respectively (symmetry codes as in Fig. 2).

Experimental

3-Chloroperbenzoic acid (77%, 247 mg, 1.10 mmol) was added in small portions to a stirred solution of ethyl 2-(5-iodo-3-methylsulfanyl-1-benzofuran-2-yl)acetate (376 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, 1:2, *v*/*v*) to afford the title compound as a colorless solid [yield 79%, m.p. 446–447 K; R_f = 0.74 (hexane-ethyl acetate, 1:2, *v*/*v*)]. 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, 0.98 Å for methyl H atoms, and 0.99 Å for methylene H atoms, and with $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic and methylene H atoms and 1.5Ueq(C) for methyl H atoms. The highest peak in the difference map is 1.36 Å from S.

Figures

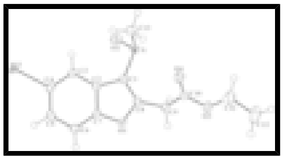


Fig. 1. The molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.

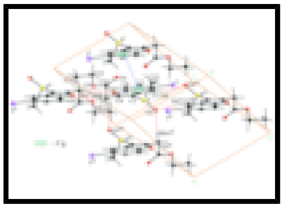


Fig. 2. π···π interactions and C—H···O hydrogen bonds (dotted lines) in the title compound. *Cg* denotes the ring centroid. [Symmetry code: (i) 1 − *x*, 2 − *y*, 1 − *z*; (ii) 2 − *x*, 2 − *y*, 1 − *z*; (iii) 2 − *x*, 2 − *y*, −*z*; (iv) 1 − *x*, 2 − *y*, −*z*.]

Ethyl 2-(5-iodo-3-methylsulfinyl-1-benzofuran-2-yl)acetate

Crystal data

$C_{13}H_{13}IO_4S$
$M_r = 392.19$
Triclinic, $P\bar{1}$
Hall symbol: -p_1
$a = 8.4311\ (4)$ Å
$b = 9.8128\ (5)$ Å
$c = 10.0299\ (5)$ Å
$\alpha = 70.629\ (1)$°
$\beta = 78.623\ (1)$°
$\gamma = 65.370\ (1)$°
$V = 709.92\ (6)$ Å^3

$Z = 2$
$F_{000} = 384$
$D_x = 1.835$ Mg m^{-3}
Mo *K*α radiation
λ = 0.71073 Å
Cell parameters from 5167 reflections
θ = 2.4–28.3°
μ = 2.41 mm^{-1}
$T = 173\ (2)$ K
Block, colorless
0.30 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite
Detector resolution: 10.0 pixels mm^{-1}
$T = 173(2)$ K
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1999)
$T_{min} = 0.561$, $T_{max} = 0.790$
6175 measured reflections

3053 independent reflections
2900 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 27.0$°
$\theta_{min} = 2.2$°
$h = -10 \rightarrow 10$
$k = -12 \rightarrow 12$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.020$
$wR(F^2) = 0.055$
$S = 1.08$

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0287P)^2 + 0.5008P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

3053 reflections	$\Delta\rho_{max} = 1.08$ e Å^{-3}
172 parameters	$\Delta\rho_{min} = -0.54$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. Spectroscopic analysis: ^{1}H NMR (CDCl$_3$, 400 MHz) δ 1.28 (t, J = 7.48 Hz, 3H), 3.06 (s, 3H), 4.04 (s, 2H), 4.20 (q, J = 7.48 Hz, 2H), 7.28 (d, J = 8.72 Hz 1H), 7.65 (dd, J = 8.72 Hz and J = 1.64 Hz, 1H), 8.27 (d, J = 1.68 Hz, 1H); ^{13}C NMR (CDCl$_3$, 100 MHz) δ 14.11, 33.40, 40.97, 62.08, 87.69, 113.85, 119.94, 126.34, 128.92, 134.43, 152.04, 153.75, 167.74; EI—MS 392[M^+].

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 > 2sigma(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}
I	0.295210 (19)	0.698038 (17)	0.404232 (16)	0.02686 (6)
S	0.72966 (7)	1.11343 (7)	0.02831 (6)	0.02383 (12)
O1	0.83624 (19)	0.96021 (18)	0.43043 (16)	0.0205 (3)
O2	0.7316 (2)	0.9883 (2)	−0.02664 (18)	0.0334 (4)
O3	0.9768 (2)	1.37474 (19)	0.22152 (19)	0.0291 (4)
O4	0.7275 (2)	1.3768 (2)	0.1722 (2)	0.0348 (4)
C1	0.7421 (3)	1.0328 (3)	0.2133 (2)	0.0196 (4)
C2	0.6560 (3)	0.9327 (2)	0.3072 (2)	0.0191 (4)
C3	0.5323 (3)	0.8778 (2)	0.2951 (2)	0.0204 (4)
H3	0.4844	0.9051	0.2073	0.024*
C4	0.4828 (3)	0.7819 (2)	0.4166 (2)	0.0210 (4)
C5	0.5511 (3)	0.7389 (3)	0.5482 (2)	0.0225 (4)
H5	0.5138	0.6717	0.6285	0.027*
C6	0.6727 (3)	0.7940 (3)	0.5611 (2)	0.0220 (4)
H6	0.7205	0.7667	0.6489	0.026*
C7	0.7207 (3)	0.8910 (2)	0.4392 (2)	0.0187 (4)
C8	0.8475 (3)	1.0452 (2)	0.2905 (2)	0.0192 (4)
C9	0.9667 (3)	1.1309 (3)	0.2571 (2)	0.0218 (4)
H9A	1.0536	1.0992	0.1798	0.026*
H9B	1.0311	1.1005	0.3415	0.026*
C10	0.8723 (3)	1.3066 (3)	0.2128 (2)	0.0224 (4)
C11	0.9013 (4)	1.5457 (3)	0.1832 (3)	0.0363 (6)
H11A	0.7912	1.5824	0.2414	0.044*
H11B	0.8755	1.5891	0.0821	0.044*

C13	0.5088 (3)	1.2521 (3)	0.0257 (3)	0.0330 (5)
H13A	0.4795	1.3090	−0.0726	0.049*
H13B	0.4963	1.3260	0.0768	0.049*
H13C	0.4296	1.1973	0.0715	0.049*
C12	1.0325 (4)	1.5972 (4)	0.2098 (5)	0.0556 (9)
H12A	1.1409	1.5599	0.1518	0.067*
H12B	1.0565	1.5540	0.3102	0.067*
H12C	0.9864	1.7115	0.1848	0.067*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I	0.02609 (9)	0.02369 (9)	0.03565 (10)	−0.01374 (6)	−0.00606 (6)	−0.00646 (6)
S	0.0260 (3)	0.0284 (3)	0.0183 (2)	−0.0125 (2)	−0.0041 (2)	−0.0038 (2)
O1	0.0213 (7)	0.0214 (7)	0.0207 (7)	−0.0097 (6)	−0.0058 (6)	−0.0037 (6)
O2	0.0392 (10)	0.0396 (10)	0.0262 (9)	−0.0137 (8)	−0.0050 (7)	−0.0150 (8)
O3	0.0268 (8)	0.0214 (8)	0.0423 (10)	−0.0115 (7)	−0.0099 (7)	−0.0055 (7)
O4	0.0282 (9)	0.0256 (9)	0.0497 (11)	−0.0111 (7)	−0.0158 (8)	−0.0006 (8)
C1	0.0198 (10)	0.0193 (10)	0.0200 (10)	−0.0072 (8)	−0.0028 (8)	−0.0053 (8)
C2	0.0202 (10)	0.0169 (10)	0.0197 (10)	−0.0045 (8)	−0.0034 (8)	−0.0066 (8)
C3	0.0219 (10)	0.0199 (10)	0.0215 (10)	−0.0066 (8)	−0.0055 (8)	−0.0081 (8)
C4	0.0203 (10)	0.0168 (10)	0.0289 (11)	−0.0069 (8)	−0.0037 (8)	−0.0090 (8)
C5	0.0244 (11)	0.0171 (10)	0.0239 (10)	−0.0079 (8)	−0.0014 (8)	−0.0034 (8)
C6	0.0249 (11)	0.0188 (10)	0.0211 (10)	−0.0062 (9)	−0.0061 (8)	−0.0044 (8)
C7	0.0170 (9)	0.0171 (10)	0.0233 (10)	−0.0052 (8)	−0.0053 (8)	−0.0066 (8)
C8	0.0203 (10)	0.0164 (10)	0.0206 (10)	−0.0062 (8)	−0.0032 (8)	−0.0048 (8)
C9	0.0205 (10)	0.0221 (10)	0.0255 (11)	−0.0093 (8)	−0.0048 (8)	−0.0066 (9)
C10	0.0257 (11)	0.0239 (11)	0.0207 (10)	−0.0127 (9)	−0.0037 (8)	−0.0045 (8)
C11	0.0343 (13)	0.0203 (12)	0.0544 (17)	−0.0102 (10)	−0.0101 (12)	−0.0070 (11)
C13	0.0313 (13)	0.0332 (13)	0.0307 (13)	−0.0072 (11)	−0.0113 (10)	−0.0056 (10)
C12	0.0454 (17)	0.0285 (15)	0.100 (3)	−0.0157 (13)	−0.0198 (18)	−0.0168 (16)

Geometric parameters (Å, °)

I—C4	2.104 (2)	C5—H5	0.950
S—O2	1.4973 (19)	C6—C7	1.385 (3)
S—C1	1.765 (2)	C6—H6	0.950
S—C13	1.790 (3)	C8—C9	1.488 (3)
O1—C7	1.377 (3)	C9—C10	1.514 (3)
O1—C8	1.382 (3)	C9—H9A	0.990
O3—C10	1.338 (3)	C9—H9B	0.990
O3—C11	1.465 (3)	C11—C12	1.490 (4)
O4—C10	1.200 (3)	C11—H11A	0.990
C1—C8	1.352 (3)	C11—H11B	0.990
C1—C2	1.445 (3)	C13—H13A	0.980
C2—C3	1.399 (3)	C13—H13B	0.980
C2—C7	1.400 (3)	C13—H13C	0.980
C3—C4	1.382 (3)	C12—H12A	0.980
C3—H3	0.950	C12—H12B	0.980

C4—C5	1.407 (3)	C12—H12C	0.980
C5—C6	1.386 (3)		

O2—S—C1	105.94 (10)	O1—C8—C9	115.37 (18)
O2—S—C13	106.48 (12)	C8—C9—C10	113.32 (18)
C1—S—C13	98.80 (11)	C8—C9—H9A	108.9
C7—O1—C8	106.37 (16)	C10—C9—H9A	108.9
C10—O3—C11	115.76 (18)	C8—C9—H9B	108.9
C8—C1—C2	107.76 (18)	C10—C9—H9B	108.9
C8—C1—S	124.73 (17)	H9A—C9—H9B	107.7
C2—C1—S	127.32 (16)	O4—C10—O3	124.1 (2)
C3—C2—C7	119.4 (2)	O4—C10—C9	126.1 (2)
C3—C2—C1	136.1 (2)	O3—C10—C9	109.77 (18)
C7—C2—C1	104.50 (18)	O3—C11—C12	107.2 (2)
C4—C3—C2	116.94 (19)	O3—C11—H11A	110.3
C4—C3—H3	121.5	C12—C11—H11A	110.3
C2—C3—H3	121.5	O3—C11—H11B	110.3
C3—C4—C5	123.1 (2)	C12—C11—H11B	110.3
C3—C4—I	118.37 (15)	H11A—C11—H11B	108.5
C5—C4—I	118.54 (16)	S—C13—H13A	109.5
C6—C5—C4	120.2 (2)	S—C13—H13B	109.5
C6—C5—H5	119.9	H13A—C13—H13B	109.5
C4—C5—H5	119.9	S—C13—H13C	109.5
C7—C6—C5	116.4 (2)	H13A—C13—H13C	109.5
C7—C6—H6	121.8	H13B—C13—H13C	109.5
C5—C6—H6	121.8	C11—C12—H12A	109.5
O1—C7—C6	125.41 (19)	C11—C12—H12B	109.5
O1—C7—C2	110.65 (18)	H12A—C12—H12B	109.5
C6—C7—C2	123.9 (2)	C11—C12—H12C	109.5
C1—C8—O1	110.72 (19)	H12A—C12—H12C	109.5
C1—C8—C9	133.9 (2)	H12B—C12—H12C	109.5

O2—S—C1—C8	135.0 (2)	C5—C6—C7—C2	−1.0 (3)
C13—S—C1—C8	−115.0 (2)	C3—C2—C7—O1	−178.10 (18)
O2—S—C1—C2	−39.3 (2)	C1—C2—C7—O1	0.7 (2)
C13—S—C1—C2	70.7 (2)	C3—C2—C7—C6	1.6 (3)
C8—C1—C2—C3	178.3 (2)	C1—C2—C7—C6	−179.7 (2)
S—C1—C2—C3	−6.6 (4)	C2—C1—C8—O1	−0.4 (2)
C8—C1—C2—C7	−0.2 (2)	S—C1—C8—O1	−175.65 (15)
S—C1—C2—C7	174.93 (16)	C2—C1—C8—C9	−179.2 (2)
C7—C2—C3—C4	−0.9 (3)	S—C1—C8—C9	5.6 (4)
C1—C2—C3—C4	−179.2 (2)	C7—O1—C8—C1	0.8 (2)
C2—C3—C4—C5	−0.1 (3)	C7—O1—C8—C9	179.83 (17)
C2—C3—C4—I	179.42 (15)	C1—C8—C9—C10	64.6 (3)
C3—C4—C5—C6	0.7 (3)	O1—C8—C9—C10	−114.1 (2)
I—C4—C5—C6	−178.86 (16)	C11—O3—C10—O4	2.4 (3)
C4—C5—C6—C7	−0.1 (3)	C11—O3—C10—C9	−179.3 (2)
C8—O1—C7—C6	179.4 (2)	C8—C9—C10—O4	−18.3 (3)
C8—O1—C7—C2	−0.9 (2)	C8—C9—C10—O3	163.43 (19)
C5—C6—C7—O1	178.62 (19)	C10—O3—C11—C12	176.0 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9B···O1^{i}	0.99	2.53	3.513 (3)	174
C9—H9A···O2^{ii}	0.99	2.20	3.190 (3)	174
C3—H3···O2^{iii}	0.95	2.48	3.391 (3)	161

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

Fig. 1

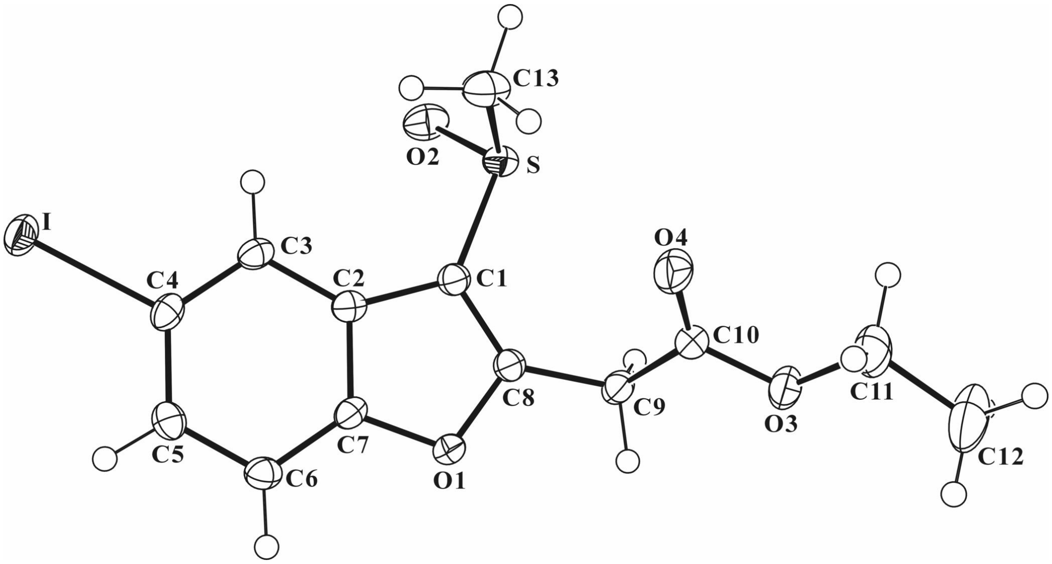
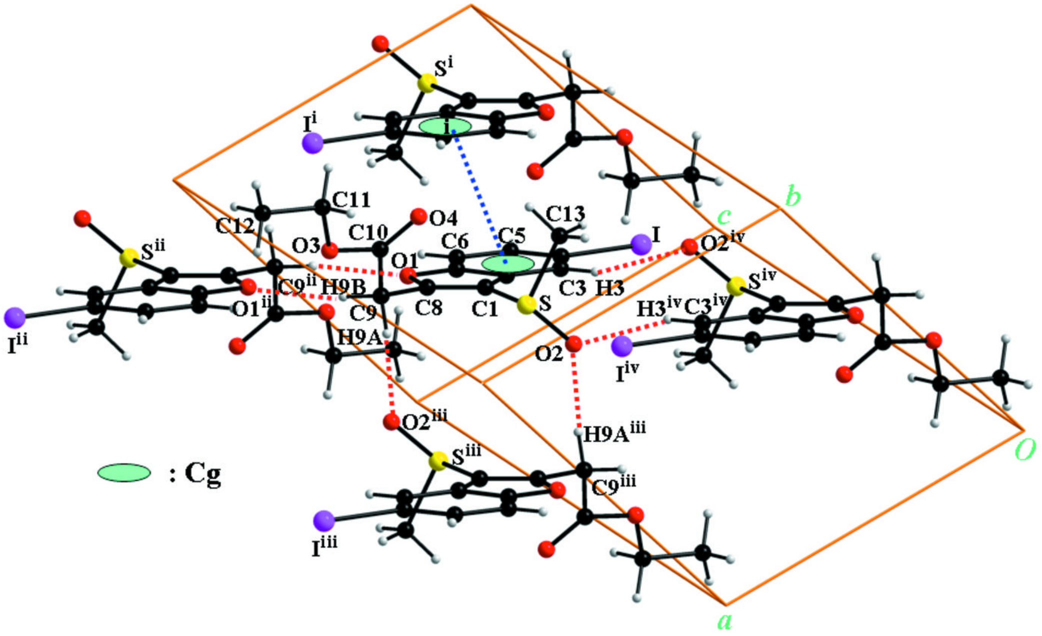


Fig. 2


S^{i}
I^{i}
C11
C12
C10
O4
O3
C6
C5
C13
I
O2^{iv}
b
c
S^{ii}
O1
C9^{ii}
H9B
C3
H3
S^{iv}
I^{ii}
O1^{ii}
C9
C8
C1
S
H3^{iv}
C3^{iv}
H9A
O2
I^{iv}
O2^{iii}
H9A^{iii}
O
: Cg
S^{iii}
C9^{iii}
I^{iii}
a