

Methyl 2-(5-iodo-7-methyl-3-methylsulfanyl-1-benzofuran-2-yl)acetate

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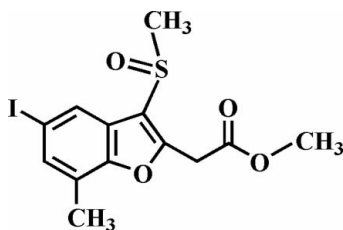
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.050; wR factor = 0.088; data-to-parameter ratio = 17.9.

There are two symmetry-independent molecules in the asymmetric unit of the title compound, $\text{C}_{13}\text{H}_{13}\text{IO}_4\text{S}$. In each molecule, the O atom and the methyl group of the methylsulfanyl substituent lie on opposite sides of the plane of the benzofuran fragment. The crystal structure is stabilized by aromatic $\pi-\pi$ interactions between the benzene and furan ring [centroid-centroid distance = $3.866(7)$ Å], and by intermolecular $\text{C}-\text{H}\cdots\pi$ interactions and a sulfanyl-sulfanyl interaction [$\text{S}\cdots\text{O} = 3.025(4)$ Å]. The crystal structure also exhibits weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds and two different $\text{I}\cdots\text{O}$ halogen bonds.

Related literature

For the crystal structures of similar alkyl 2-(5-iodo-3-methylsulfanyl-1-benzofuran-2-yl)acetate derivatives, see: Choi *et al.* (2008*a,b*). For halogen bonding, see: Politzer *et al.* (2007). For carbonyl-carbonyl interactions, see: Allen *et al.* (1998).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{13}\text{IO}_4\text{S}$ $c = 17.845(1)$ Å
 $M_r = 392.19$ $\alpha = 77.701(1)^\circ$
 Triclinic, $P\bar{1}$ $\beta = 88.074(1)^\circ$
 $a = 7.5424(4)$ Å $\gamma = 88.229(1)^\circ$
 $b = 11.2177(6)$ Å $V = 1473.92(14)$ Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 2.32$ mm⁻¹

$T = 293$ K
 $0.30 \times 0.20 \times 0.10$ mm

Data collection

Bruker SMART CCD diffractometer 12617 measured reflections
 Absorption correction: multi-scan (SADABS; Sheldrick, 1999) 6248 independent reflections
 $T_{\min} = 0.574$, $T_{\max} = 0.790$ 5328 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.050$ 349 parameters
 $wR(F^2) = 0.088$ H-atom parameters constrained
 $S = 1.18$ $\Delta\rho_{\text{max}} = 1.15$ e Å⁻³
 6248 reflections $\Delta\rho_{\text{min}} = -1.46$ e Å⁻³

Table 1

Selected interatomic distances (Å).

$\text{I1}\cdots\text{O7}^{\text{i}}$	3.300 (3)	$\text{I2}\cdots\text{O3}^{\text{ii}}$	3.264 (3)
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Symmetry codes: (i) $x, y - 1, z + 1$; (ii) $x + 1, y, z$.

Table 2

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C5}-\text{H5}\cdots\text{O3}^{\text{iii}}$	0.93	2.59	3.430 (6)	150
$\text{C11}-\text{H11B}\cdots\text{O8}$	0.96	2.51	3.254 (6)	134
$\text{C12}-\text{H12C}\cdots\text{O4}^{\text{iv}}$	0.96	2.58	3.489 (6)	158
$\text{C16}-\text{H16}\cdots\text{O8}^{\text{v}}$	0.93	2.48	3.373 (6)	160
$\text{C18}-\text{H18}\cdots\text{O4}^{\text{vi}}$	0.93	2.44	3.297 (6)	153
$\text{C22}-\text{H22B}\cdots\text{O8}^{\text{vii}}$	0.97	2.27	3.215 (6)	164
$\text{C12}-\text{H12A}\cdots\text{Cg1}^{\text{iii}}$	0.96	2.85	3.734 (7)	153
$\text{C24}-\text{H24A}\cdots\text{Cg3}^{\text{viii}}$	0.96	2.73	3.574 (7)	147

Symmetry codes: (iii) $-x + 1, -y, -z + 1$; (iv) $-x + 2, -y, -z + 1$; (v) $-x + 2, -y + 1, -z$; (vi) $-x + 2, -y + 1, -z + 1$; (vii) $-x + 1, -y + 1, -z$; (viii) $-x + 1, -y + 2, -z$. Cg1 and Cg3 are the centroids of the C2-C7 benzene ring and the C14/C15/C20/O5/C21 furan ring, respectively.

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: GW2062).

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supplementary materials

Acta Cryst. (2009). E65, o1240 [doi:10.1107/S1600536809016298]

Methyl 2-(5-iodo-7-methyl-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-iodo-3-methylsulfinyl-1-benzofuran-2-yl)acetate analogues, *viz.* isopropyl 2-(5-iodo-3-methylsulfinyl-1-benzofuran-2-yl)acetate (Choi *et al.*, 2008*a*) and isopropyl 2-(5-iodo-7-methyl-3-methylsulfinyl-1-benzofuran-2-yl)acetate (Choi *et al.*, 2008*b*). Here we report the crystal structure of the title compound, methyl 2-(5-iodo-7-methyl-3-methylsulfinyl-1-benzofuran-2-yl)acetate (Fig. 1). The benzofuran unit is essentially planar, with a mean deviation of 0.017 (4) Å for two independent molecules, from the least-squares plane defined by the nine constituent atoms.

The molecular packing (Fig. 2 & 3) is stabilized by aromatic π – π interactions between the benzene ring and the furan ring of adjacent benzofuran units, with a $Cg1 \cdots Cg2^{ix}$ distance of 3.866 (7) Å ($Cg1$ and $Cg2$ are the centroids of the C2–C7 benzene ring and the C1/C2/C7/O1/C8 furan ring; symmetry code as in Fig. 3). The crystal packing is further stabilized by C—H $\cdots\pi$ interactions (Table 2 and Fig. 3); the first between the methyl H atom and the benzene ring of the benzofuran unit, *i.e.* C12—H12A $\cdots Cg1^{iii}$, the second between the methyl H atom and the furan ring of the benzofuran unit, *i.e.* C24—H24A $\cdots Cg3^{viii}$, respectively ($Cg3$ is the centroid of the C14/C15/C20/O5/C21 furan ring; symmetry code as in Fig. 3), and by an intermolecular sulfinyl–sulfinyl interaction interpreted as similar to a type-II carbonyl–carbonyl interaction (Allen *et al.*, 1998), with S1 $\cdots O4^{xi}$ and O4 $\cdots S1^{xi}$ distance of 3.025 (4) Å (Fig. 3; symmetry code as in Fig. 3). In addition, the crystal packing exhibits weak intermolecular C—H $\cdots O$ hydrogen bonds (Fig. 2 and Table 2). In crystal structure, there are two different I $\cdots O$ halogen bonds (Politzer *et al.*, 2007) between the two independent iodine atoms and the oxygen atoms of neighbouring C=O units (Fig. 2 and Table 1).

Experimental

77% 3-chloroperoxybenzoic acid (123 mg, 0.55 mmol) was added in small portions to a stirred solution of methyl 2-(5-iodo-7-methyl-3-methylsulfinyl-1-benzofuran-2-yl)acetate (202 mg, 0.5 mmol) in dichloromethane (30 ml) at 273 K. After being stirred 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 (ethyl acetate) to afford the title compound as a colorless solid [yield 83%, m.p. 414–415 K; R_f = 0.63 (ethyl acetate)]. Single crystals suitable for X-ray diffraction were prepared by evaporation of a solution of the title compound in benzene at room temperature. Spectroscopic analysis: 1H NMR ($CDCl_3$, 400 MHz) δ 2.46 (s, 3H), 3.05 (s, 3H), 3.76 (s, 3H), 4.07 (s, 2H), 7.49 (s, 1H), 8.07 (s, 1H); EI—MS 392 [M^+].

Refinement

All H atoms were geometrically positioned and refined using a riding model, with C—H = 0.93 Å for the aryl, 0.97 Å for the methylene, and 0.96 Å for the methyl H atoms. $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for the aryl and methylene H atoms, and $1.5U_{\text{eq}}(\text{C})$ for methyl H atoms.

Figures

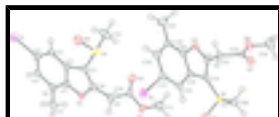


Fig. 1. The molecular structure of the title compound, showing displacement ellipsoids drawn at the 30% probability level.

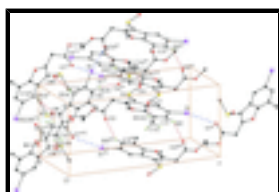


Fig. 2. C—H...O hydrogen bond and I...O halogen bond (dotted lines) in the title compound [Symmetry code: (i) $x, y - 1, z + 1$; (ii) $x + 1, y, z$; (iii) $-x + 1, -y, -z + 1$; (iv) $-x + 2, -y, -z + 1$; (v) $-x + 2, -y + 1, -z$; (vi) $2 - x, 1 - y, 1 - z$; (vii) $-x + 1, -y + 1, -z$.]

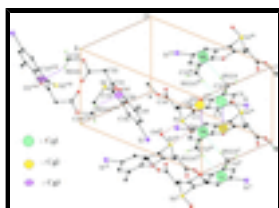


Fig. 3. π — π , C—H... π , and S...O interactions (dotted lines) in the title compound. Cg denotes the ring centroids [Symmetry code: (iii) $-x + 1, -y, -z + 1$; (viii) $-x + 1, -y + 2, -z$; (ix) $-x + 2, -y, -z + 1$; (x) $x + 1, y, z$; (xi) $-x + 2, -y + 1, -z + 1$.]

Methyl 2-(5-iodo-7-methyl-3-methylsulfinyl-1-benzofuran-2-yl)acetate

Crystal data

$\text{C}_{13}\text{H}_{13}\text{IO}_4\text{S}$

$M_r = 392.19$

Triclinic, $P\bar{1}$

Hall symbol: -p 1

$a = 7.5424$ (4) Å

$b = 11.2177$ (6) Å

$c = 17.845$ (1) Å

$\alpha = 77.701$ (1)°

$\beta = 88.074$ (1)°

$\gamma = 88.229$ (1)°

$V = 1473.92$ (14) Å³

$Z = 4$

$F_{000} = 768$

$D_x = 1.767$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 6307 reflections

$\theta = 2.3$ – 28.2 °

$\mu = 2.32$ mm⁻¹

$T = 293$ K

Block, colorless

$0.30 \times 0.20 \times 0.10$ mm

Data collection

Bruker SMART CCD
diffractometer

6248 independent reflections

Radiation source: fine-focus sealed tube	5328 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.027$
Detector resolution: 10.0 pixels mm^{-1}	$\theta_{\text{max}} = 27.0^\circ$
$T = 293$ K	$\theta_{\text{min}} = 1.9^\circ$
φ and ω scans	$h = -9 \rightarrow 9$
Absorption correction: multi-scan (SADABS; Sheldrick, 1999)	$k = -14 \rightarrow 14$
$T_{\text{min}} = 0.574$, $T_{\text{max}} = 0.790$	$l = -22 \rightarrow 22$
12617 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.050$	H-atom parameters constrained
$wR(F^2) = 0.088$	$w = 1/[\sigma^2(F_o^2) + (0.0184P)^2 + 4.3135P]$
$S = 1.18$	where $P = (F_o^2 + 2F_c^2)/3$
6248 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
349 parameters	$\Delta\rho_{\text{max}} = 1.15 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -1.46 \text{ e } \text{\AA}^{-3}$
	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 $F^2 > \sigma(F^2)$ 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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
I1	0.66948 (5)	-0.00958 (3)	0.780861 (19)	0.03974 (11)
S1	0.92033 (15)	0.36686 (11)	0.47095 (7)	0.0253 (3)
O1	0.7992 (4)	0.0463 (3)	0.43207 (17)	0.0234 (7)
O2	0.7940 (4)	0.2574 (3)	0.20886 (18)	0.0285 (8)
O3	0.6320 (5)	0.3131 (3)	0.30365 (19)	0.0340 (8)
O4	1.0056 (5)	0.3653 (3)	0.5448 (2)	0.0343 (8)
C1	0.8593 (6)	0.2142 (4)	0.4747 (3)	0.0210 (9)
C2	0.7987 (6)	0.1252 (4)	0.5400 (3)	0.0193 (9)
C3	0.7743 (6)	0.1181 (4)	0.6187 (3)	0.0217 (10)

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H3	0.8012	0.1827	0.6411	0.026*
C4	0.7085 (6)	0.0108 (4)	0.6616 (3)	0.0226 (10)
C5	0.6671 (6)	-0.0880 (4)	0.6299 (3)	0.0249 (10)
H5	0.6212	-0.1579	0.6614	0.030*
C6	0.6938 (6)	-0.0833 (4)	0.5515 (3)	0.0260 (10)
C7	0.7615 (5)	0.0234 (4)	0.5097 (2)	0.0192 (9)
C8	0.8613 (6)	0.1634 (4)	0.4126 (3)	0.0236 (10)
C9	0.9133 (6)	0.2074 (5)	0.3309 (3)	0.0304 (12)
H9A	0.9629	0.1393	0.3109	0.037*
H9B	1.0056	0.2669	0.3274	0.037*
C10	0.7613 (6)	0.2650 (4)	0.2812 (3)	0.0229 (10)
C11	0.6621 (7)	0.3127 (5)	0.1547 (3)	0.0333 (12)
H11A	0.5579	0.2640	0.1625	0.050*
H11B	0.7086	0.3177	0.1034	0.050*
H11C	0.6319	0.3933	0.1621	0.050*
C12	0.6516 (7)	-0.1891 (5)	0.5159 (3)	0.0331 (12)
H12A	0.5628	-0.1639	0.4781	0.050*
H12B	0.6079	-0.2550	0.5550	0.050*
H12C	0.7571	-0.2158	0.4920	0.050*
C13	0.7001 (7)	0.4299 (5)	0.4797 (4)	0.0403 (14)
H13A	0.6444	0.3883	0.5267	0.060*
H13B	0.6317	0.4198	0.4371	0.060*
H13C	0.7069	0.5151	0.4798	0.060*
I2	1.27204 (4)	0.47039 (3)	0.246411 (19)	0.02932 (10)
S2	0.68848 (15)	0.62050 (11)	-0.02104 (6)	0.0208 (2)
O5	0.5483 (4)	0.7323 (3)	0.16778 (17)	0.0228 (7)
O6	0.2057 (4)	0.9129 (3)	-0.0229 (2)	0.0348 (9)
O7	0.5006 (4)	0.8964 (3)	-0.04426 (19)	0.0307 (8)
O8	0.7522 (4)	0.4897 (3)	-0.01091 (18)	0.0288 (8)
C14	0.6717 (6)	0.6561 (4)	0.0710 (2)	0.0192 (9)
C15	0.7950 (6)	0.6310 (4)	0.1331 (2)	0.0201 (9)
C16	0.9597 (6)	0.5710 (4)	0.1474 (3)	0.0226 (10)
H16	1.0203	0.5374	0.1103	0.027*
C17	1.0283 (6)	0.5640 (4)	0.2189 (3)	0.0241 (10)
C18	0.9410 (7)	0.6153 (4)	0.2756 (3)	0.0287 (11)
H18	0.9940	0.6092	0.3227	0.034*
C19	0.7787 (7)	0.6744 (5)	0.2632 (3)	0.0291 (11)
C20	0.7105 (6)	0.6795 (4)	0.1915 (3)	0.0221 (10)
C21	0.5275 (6)	0.7141 (4)	0.0946 (2)	0.0189 (9)
C22	0.3549 (6)	0.7575 (4)	0.0601 (3)	0.0232 (10)
H22A	0.2778	0.7819	0.0991	0.028*
H22B	0.3000	0.6902	0.0442	0.028*
C23	0.3682 (6)	0.8633 (4)	-0.0081 (3)	0.0215 (10)
C24	0.1942 (8)	1.0119 (5)	-0.0898 (3)	0.0457 (15)
H24A	0.2691	1.0767	-0.0837	0.069*
H24B	0.0737	1.0418	-0.0953	0.069*
H24C	0.2322	0.9828	-0.1347	0.069*
C25	0.6774 (8)	0.7295 (6)	0.3224 (3)	0.0482 (16)
H25A	0.7436	0.7163	0.3686	0.072*

H25B	0.5643	0.6917	0.3333	0.072*
H25C	0.6600	0.8155	0.3031	0.072*
C26	0.8772 (6)	0.7101 (5)	-0.0563 (3)	0.0284 (11)
H26A	0.9763	0.6818	-0.0243	0.043*
H26B	0.8507	0.7941	-0.0556	0.043*
H26C	0.9061	0.7024	-0.1079	0.043*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0579 (3)	0.0384 (2)	0.02000 (17)	0.01037 (17)	0.00379 (15)	-0.00193 (14)
S1	0.0221 (6)	0.0216 (6)	0.0315 (6)	-0.0007 (5)	-0.0032 (5)	-0.0038 (5)
O1	0.0225 (16)	0.0297 (18)	0.0195 (16)	0.0001 (14)	-0.0032 (13)	-0.0085 (14)
O2	0.0289 (18)	0.038 (2)	0.0172 (16)	0.0048 (15)	-0.0014 (14)	-0.0026 (14)
O3	0.0275 (19)	0.045 (2)	0.0254 (18)	0.0095 (16)	0.0018 (15)	-0.0014 (16)
O4	0.040 (2)	0.030 (2)	0.0317 (19)	-0.0043 (16)	-0.0048 (16)	-0.0033 (16)
C1	0.019 (2)	0.020 (2)	0.024 (2)	-0.0017 (18)	-0.0020 (18)	-0.0055 (19)
C2	0.016 (2)	0.017 (2)	0.024 (2)	0.0020 (17)	-0.0005 (17)	-0.0028 (18)
C3	0.024 (2)	0.021 (2)	0.021 (2)	0.0042 (18)	-0.0073 (18)	-0.0058 (18)
C4	0.020 (2)	0.029 (3)	0.020 (2)	0.0078 (19)	-0.0036 (18)	-0.0077 (19)
C5	0.022 (2)	0.018 (2)	0.032 (3)	0.0036 (18)	0.0000 (19)	-0.0002 (19)
C6	0.018 (2)	0.023 (2)	0.038 (3)	0.0060 (19)	-0.005 (2)	-0.009 (2)
C7	0.014 (2)	0.028 (2)	0.016 (2)	0.0004 (18)	-0.0050 (16)	-0.0048 (18)
C8	0.016 (2)	0.025 (2)	0.028 (2)	0.0031 (18)	-0.0052 (18)	-0.001 (2)
C9	0.021 (2)	0.048 (3)	0.020 (2)	0.005 (2)	0.0014 (19)	-0.002 (2)
C10	0.018 (2)	0.027 (3)	0.021 (2)	-0.0007 (19)	-0.0011 (18)	0.0006 (19)
C11	0.032 (3)	0.044 (3)	0.021 (2)	0.001 (2)	-0.005 (2)	-0.002 (2)
C12	0.026 (3)	0.028 (3)	0.048 (3)	0.001 (2)	-0.004 (2)	-0.015 (2)
C13	0.022 (3)	0.023 (3)	0.075 (4)	0.002 (2)	-0.007 (3)	-0.010 (3)
I2	0.02201 (16)	0.03174 (19)	0.03059 (18)	0.00357 (13)	-0.00415 (13)	0.00141 (14)
S2	0.0241 (6)	0.0247 (6)	0.0145 (5)	0.0012 (5)	-0.0013 (4)	-0.0061 (4)
O5	0.0260 (17)	0.0245 (17)	0.0187 (16)	0.0080 (13)	-0.0023 (13)	-0.0074 (13)
O6	0.0237 (18)	0.032 (2)	0.043 (2)	0.0059 (15)	-0.0063 (16)	0.0050 (16)
O7	0.0295 (19)	0.033 (2)	0.0262 (18)	0.0012 (15)	0.0048 (15)	0.0008 (15)
O8	0.037 (2)	0.0257 (18)	0.0240 (17)	0.0027 (15)	0.0050 (15)	-0.0080 (14)
C14	0.022 (2)	0.020 (2)	0.015 (2)	-0.0008 (18)	0.0016 (17)	-0.0042 (17)
C15	0.026 (2)	0.019 (2)	0.016 (2)	-0.0026 (18)	0.0017 (18)	-0.0044 (17)
C16	0.024 (2)	0.023 (2)	0.020 (2)	0.0008 (19)	0.0015 (18)	-0.0059 (19)
C17	0.022 (2)	0.020 (2)	0.027 (2)	0.0006 (19)	-0.0035 (19)	0.0011 (19)
C18	0.036 (3)	0.030 (3)	0.020 (2)	0.004 (2)	-0.011 (2)	-0.005 (2)
C19	0.041 (3)	0.031 (3)	0.016 (2)	0.010 (2)	-0.006 (2)	-0.009 (2)
C20	0.027 (2)	0.020 (2)	0.018 (2)	0.0035 (19)	-0.0024 (18)	-0.0028 (18)
C21	0.024 (2)	0.019 (2)	0.014 (2)	-0.0021 (18)	-0.0015 (17)	-0.0018 (17)
C22	0.022 (2)	0.030 (3)	0.017 (2)	-0.0006 (19)	0.0015 (18)	-0.0037 (19)
C23	0.027 (2)	0.015 (2)	0.026 (2)	0.0066 (19)	0.001 (2)	-0.0125 (19)
C24	0.037 (3)	0.038 (3)	0.053 (4)	0.011 (3)	-0.011 (3)	0.011 (3)
C25	0.057 (4)	0.066 (4)	0.027 (3)	0.029 (3)	-0.011 (3)	-0.023 (3)
C26	0.031 (3)	0.032 (3)	0.021 (2)	-0.004 (2)	0.008 (2)	-0.002 (2)

supplementary materials

Geometric parameters (Å, °)

I1—C4	2.103 (4)	I2—C17	2.108 (4)
I1—O7 ⁱ	3.300 (3)	I2—O3 ⁱⁱⁱ	3.264 (3)
S1—O4	1.482 (4)	S2—O8	1.505 (3)
S1—C1	1.773 (5)	S2—C14	1.771 (4)
S1—C13	1.800 (5)	S2—C26	1.786 (5)
S1—O4 ⁱⁱ	3.025 (4)	O5—C21	1.379 (5)
O1—C7	1.375 (5)	O5—C20	1.382 (5)
O1—C8	1.378 (6)	O6—C23	1.341 (5)
O2—C10	1.326 (6)	O6—C24	1.451 (6)
O2—C11	1.442 (6)	O7—C23	1.196 (5)
O3—C10	1.198 (5)	C14—C21	1.351 (6)
C1—C8	1.349 (6)	C14—C15	1.446 (6)
C1—C2	1.435 (6)	C15—C20	1.400 (6)
C2—C3	1.397 (6)	C15—C16	1.400 (6)
C2—C7	1.403 (6)	C16—C17	1.379 (6)
C3—C4	1.378 (6)	C16—H16	0.9300
C3—H3	0.9300	C17—C18	1.403 (7)
C4—C5	1.395 (7)	C18—C19	1.376 (7)
C5—C6	1.397 (7)	C18—H18	0.9300
C5—H5	0.9300	C19—C20	1.385 (6)
C6—C7	1.371 (7)	C19—C25	1.509 (7)
C6—C12	1.507 (7)	C21—C22	1.482 (6)
C8—C9	1.481 (6)	C22—C23	1.511 (6)
C9—C10	1.516 (6)	C22—H22A	0.9700
C9—H9A	0.9700	C22—H22B	0.9700
C9—H9B	0.9700	C24—H24A	0.9600
C11—H11A	0.9600	C24—H24B	0.9600
C11—H11B	0.9600	C24—H24C	0.9600
C11—H11C	0.9600	C25—H25A	0.9600
C12—H12A	0.9600	C25—H25B	0.9600
C12—H12B	0.9600	C25—H25C	0.9600
C12—H12C	0.9600	C26—H26A	0.9600
C13—H13A	0.9600	C26—H26B	0.9600
C13—H13B	0.9600	C26—H26C	0.9600
C13—H13C	0.9600		
I1...O7 ⁱ	3.300 (3)	I2...O3 ⁱⁱⁱ	3.264 (3)
C4—I1—O7 ⁱ	160.08 (15)	H13B—C13—H13C	109.5
O4—S1—C1	105.7 (2)	C17—I2—O3 ⁱⁱⁱ	174.78 (14)
O4—S1—C13	106.0 (3)	O8—S2—C14	107.7 (2)
C1—S1—C13	97.4 (2)	O8—S2—C26	105.5 (2)
O4—S1—O4 ⁱⁱ	78.94 (17)	C14—S2—C26	97.6 (2)
C1—S1—O4 ⁱⁱ	174.59 (17)	C21—O5—C20	106.4 (3)
C13—S1—O4 ⁱⁱ	78.48 (18)	C23—O6—C24	115.7 (4)
C7—O1—C8	106.4 (3)	C21—C14—C15	107.9 (4)

C10—O2—C11	116.0 (4)	C21—C14—S2	122.2 (3)
C8—C1—C2	108.3 (4)	C15—C14—S2	130.0 (3)
C8—C1—S1	123.4 (4)	C20—C15—C16	118.7 (4)
C2—C1—S1	128.3 (3)	C20—C15—C14	104.4 (4)
C3—C2—C7	119.1 (4)	C16—C15—C14	136.8 (4)
C3—C2—C1	136.7 (4)	C17—C16—C15	117.0 (4)
C7—C2—C1	104.2 (4)	C17—C16—H16	121.5
C4—C3—C2	116.8 (4)	C15—C16—H16	121.5
C4—C3—H3	121.6	C16—C17—C18	122.7 (4)
C2—C3—H3	121.6	C16—C17—I2	119.5 (3)
C3—C4—C5	123.2 (4)	C18—C17—I2	117.8 (3)
C3—C4—I1	119.8 (3)	C19—C18—C17	121.4 (4)
C5—C4—I1	117.1 (3)	C19—C18—H18	119.3
C4—C5—C6	120.7 (4)	C17—C18—H18	119.3
C4—C5—H5	119.6	C18—C19—C20	115.2 (4)
C6—C5—H5	119.6	C18—C19—C25	123.8 (4)
C7—C6—C5	115.5 (4)	C20—C19—C25	121.0 (5)
C7—C6—C12	122.6 (5)	O5—C20—C19	124.6 (4)
C5—C6—C12	121.8 (5)	O5—C20—C15	110.5 (4)
C6—C7—O1	124.8 (4)	C19—C20—C15	124.9 (4)
C6—C7—C2	124.6 (4)	C14—C21—O5	110.8 (4)
O1—C7—C2	110.6 (4)	C14—C21—C22	134.1 (4)
C1—C8—O1	110.5 (4)	O5—C21—C22	115.1 (4)
C1—C8—C9	133.7 (5)	C21—C22—C23	114.2 (4)
O1—C8—C9	115.8 (4)	C21—C22—H22A	108.7
C8—C9—C10	113.7 (4)	C23—C22—H22A	108.7
C8—C9—H9A	108.8	C21—C22—H22B	108.7
C10—C9—H9A	108.8	C23—C22—H22B	108.7
C8—C9—H9B	108.8	H22A—C22—H22B	107.6
C10—C9—H9B	108.8	O7—C23—O6	125.1 (4)
H9A—C9—H9B	107.7	O7—C23—C22	126.0 (4)
O3—C10—O2	124.9 (4)	O6—C23—C22	108.8 (4)
O3—C10—C9	124.9 (4)	O6—C24—H24A	109.5
O2—C10—C9	110.2 (4)	O6—C24—H24B	109.5
O2—C11—H11A	109.5	H24A—C24—H24B	109.5
O2—C11—H11B	109.5	O6—C24—H24C	109.5
H11A—C11—H11B	109.5	H24A—C24—H24C	109.5
O2—C11—H11C	109.5	H24B—C24—H24C	109.5
H11A—C11—H11C	109.5	C19—C25—H25A	109.5
H11B—C11—H11C	109.5	C19—C25—H25B	109.5
C6—C12—H12A	109.5	H25A—C25—H25B	109.5
C6—C12—H12B	109.5	C19—C25—H25C	109.5
H12A—C12—H12B	109.5	H25A—C25—H25C	109.5
C6—C12—H12C	109.5	H25B—C25—H25C	109.5
H12A—C12—H12C	109.5	S2—C26—H26A	109.5
H12B—C12—H12C	109.5	S2—C26—H26B	109.5
S1—C13—H13A	109.5	H26A—C26—H26B	109.5
S1—C13—H13B	109.5	S2—C26—H26C	109.5
H13A—C13—H13B	109.5	H26A—C26—H26C	109.5

supplementary materials

S1—C13—H13C	109.5	H26B—C26—H26C	109.5
H13A—C13—H13C	109.5		
O4—S1—C1—C8	-146.0 (4)	C8—C9—C10—O2	-155.0 (4)
C13—S1—C1—C8	105.1 (4)	O8—S2—C14—C21	-133.1 (4)
O4—S1—C1—C2	35.0 (5)	C26—S2—C14—C21	117.9 (4)
C13—S1—C1—C2	-74.0 (5)	O8—S2—C14—C15	45.9 (5)
C8—C1—C2—C3	176.9 (5)	C26—S2—C14—C15	-63.1 (5)
S1—C1—C2—C3	-4.0 (8)	C21—C14—C15—C20	-0.5 (5)
C8—C1—C2—C7	-2.0 (5)	S2—C14—C15—C20	-179.6 (4)
S1—C1—C2—C7	177.2 (3)	C21—C14—C15—C16	176.6 (5)
C7—C2—C3—C4	-2.1 (6)	S2—C14—C15—C16	-2.5 (8)
C1—C2—C3—C4	179.2 (5)	C20—C15—C16—C17	-0.1 (6)
C2—C3—C4—C5	0.1 (7)	C14—C15—C16—C17	-176.9 (5)
C2—C3—C4—I1	179.4 (3)	C15—C16—C17—C18	-0.9 (7)
O7 ⁱ —I1—C4—C3	150.8 (3)	C15—C16—C17—I2	178.1 (3)
O7 ⁱ —I1—C4—C5	-29.9 (6)	C16—C17—C18—C19	1.1 (8)
C3—C4—C5—C6	1.0 (7)	I2—C17—C18—C19	-177.9 (4)
I1—C4—C5—C6	-178.2 (3)	C17—C18—C19—C20	-0.3 (7)
C4—C5—C6—C7	-0.1 (6)	C17—C18—C19—C25	179.0 (5)
C4—C5—C6—C12	179.5 (4)	C21—O5—C20—C19	-177.8 (5)
C5—C6—C7—O1	179.3 (4)	C21—O5—C20—C15	1.7 (5)
C12—C6—C7—O1	-0.3 (7)	C18—C19—C20—O5	178.6 (4)
C5—C6—C7—C2	-2.0 (7)	C25—C19—C20—O5	-0.6 (8)
C12—C6—C7—C2	178.4 (4)	C18—C19—C20—C15	-0.7 (8)
C8—O1—C7—C6	179.0 (4)	C25—C19—C20—C15	-180.0 (5)
C8—O1—C7—C2	0.1 (5)	C16—C15—C20—O5	-178.5 (4)
C3—C2—C7—C6	3.2 (7)	C14—C15—C20—O5	-0.7 (5)
C1—C2—C7—C6	-177.7 (4)	C16—C15—C20—C19	0.9 (7)
C3—C2—C7—O1	-178.0 (4)	C14—C15—C20—C19	178.7 (5)
C1—C2—C7—O1	1.1 (5)	C15—C14—C21—O5	1.6 (5)
C2—C1—C8—O1	2.2 (5)	S2—C14—C21—O5	-179.2 (3)
S1—C1—C8—O1	-177.0 (3)	C15—C14—C21—C22	-176.3 (5)
C2—C1—C8—C9	-178.5 (5)	S2—C14—C21—C22	2.9 (7)
S1—C1—C8—C9	2.3 (8)	C20—O5—C21—C14	-2.0 (5)
C7—O1—C8—C1	-1.4 (5)	C20—O5—C21—C22	176.3 (4)
C7—O1—C8—C9	179.1 (4)	C14—C21—C22—C23	-68.7 (7)
C1—C8—C9—C10	-92.0 (6)	O5—C21—C22—C23	113.6 (4)
O1—C8—C9—C10	87.3 (5)	C24—O6—C23—O7	1.9 (7)
C11—O2—C10—O3	0.4 (7)	C24—O6—C23—C22	-176.7 (4)
C11—O2—C10—C9	-177.9 (4)	C21—C22—C23—O7	15.9 (7)
C8—C9—C10—O3	26.6 (7)	C21—C22—C23—O6	-165.5 (4)

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C5—H5 ^{iv} —O3 ^{iv}	0.93	2.59	3.430 (6)	150
C11—H11B ^{iv} —O8	0.96	2.51	3.254 (6)	134

C12—H12C···O4 ^v	0.96	2.58	3.489 (6)	158
C16—H16···O8 ^{vi}	0.93	2.48	3.373 (6)	160
C18—H18···O4 ⁱⁱ	0.93	2.44	3.297 (6)	153
C22—H22B···O8 ^{vii}	0.97	2.27	3.215 (6)	164
C12—H12A···Cg1 ^{iv}	0.96	2.85	3.734 (7)	153
C24—H24A···Cg3 ^{viii}	0.96	2.73	3.574 (7)	147

Symmetry codes: (iv) $-x+1, -y, -z+1$; (v) $-x+2, -y, -z+1$; (vi) $-x+2, -y+1, -z$; (ii) $-x+2, -y+1, -z+1$; (vii) $-x+1, -y+1, -z$; (viii) $-x+1, -y+2, -z$.

Fig. 1

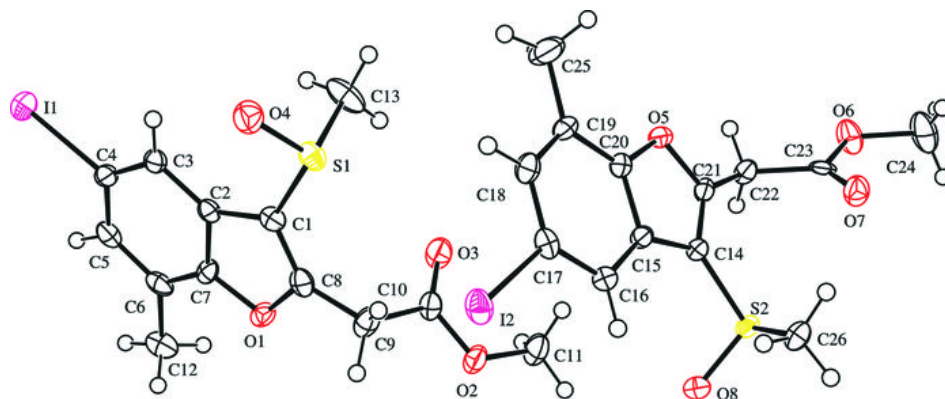


Fig. 2

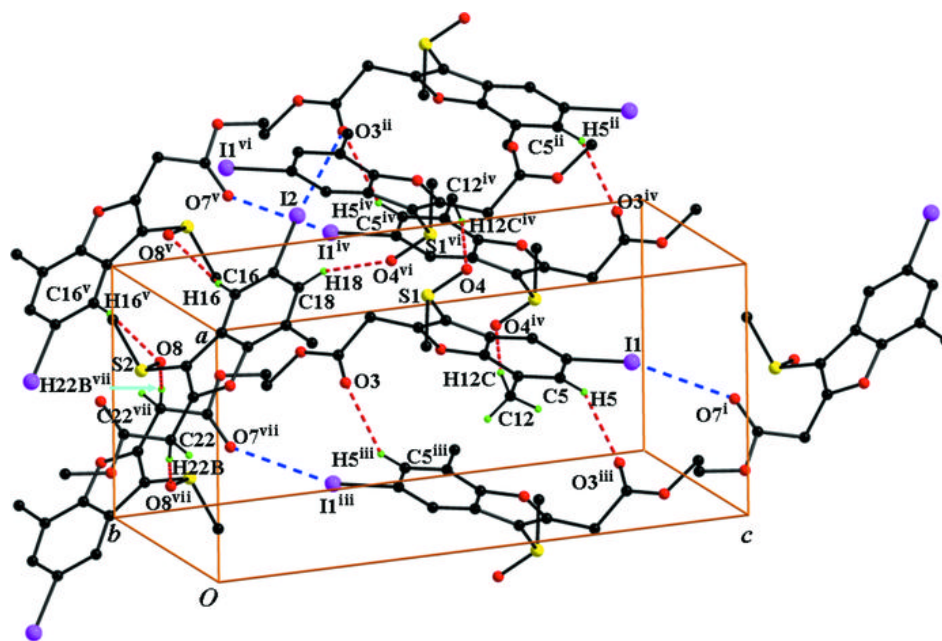


Fig. 3

