

5-Iodo-2-methyl-3-methylsulfinyl-1-benzofuran

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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.028; wR factor = 0.066; data-to-parameter ratio = 21.1.

The title compound, $\text{C}_{10}\text{H}_9\text{IO}_2\text{S}$, was prepared by the oxidation of 5-iodo-2-methyl-3-methylsulfonyl-1-benzofuran 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 short $\text{C}-\text{H}\cdots\pi$ interactions between the methyl H atoms and the benzene ring of the benzofuran system, and by an $\text{I}\cdots\text{O}$ halogen bond of 3.139 (2) Å and a nearly linear $\text{C}-\text{I}\cdots\text{O}$ angle of 164.13 (9)°. Additionally, the stacked molecules exhibit an intermolecular sulfinyl-sulfinyl interaction [3.190 (2) Å].

Related literature

For the crystal structures of similar 5-iodobenzofuran compounds, see: Choi *et al.* (2007*a,b*). For a review of halogen bonding, see: Politzer *et al.* (2007). For a review of carbonyl-carbonyl interactions, see: Allen *et al.* (1998).



Experimental

Crystal data

$\text{C}_{10}\text{H}_9\text{IO}_2\text{S}$	$\gamma = 97.620$ (2)°
$M_r = 320.13$	$V = 548.52$ (5) Å ³
Triclinic, $P\bar{1}$	$Z = 2$
$a = 8.1521$ (4) Å	Mo $K\alpha$ radiation
$b = 8.2671$ (4) Å	$\mu = 3.08$ mm ⁻¹
$c = 8.6870$ (4) Å	$T = 298$ (2) K
$\alpha = 104.585$ (2)°	$0.41 \times 0.35 \times 0.20$ mm
$\beta = 99.920$ (2)°	

Data collection

Bruker SMART CCD diffractometer	10576 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2000)	2695 independent reflections
$T_{\min} = 0.301$, $T_{\max} = 0.538$	2448 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.017$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.028$	128 parameters
$wR(F^2) = 0.066$	H-atom parameters constrained
$S = 1.08$	$\Delta\rho_{\text{max}} = 1.31$ e Å ⁻³
2695 reflections	$\Delta\rho_{\text{min}} = -0.73$ e Å ⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C9}-\text{H9B}\cdots\text{Cg}^i$	0.96	2.72	3.527 (3)	142

Symmetry code: (i) $-x + 1, -y, -z + 1$. 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, 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: SJ2426).

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supplementary materials

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5-Iodo-2-methyl-3-methylsulfinyl-1-benzofuran

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Comment

As part of our ongoing studies of the synthesis and structure of 5-iodo-1-benzofuran derivatives, we have recently described the crystal structures of 5-iodo-3-methylsulfinyl-2-phenyl-1-benzofuran (Choi *et al.*, 2007*a*) and 5-iodo-2-methyl-3-phenylsulfinyl-1-benzofuran (Choi *et al.*, 2007*b*). Herein we report the crystal and molecular structure of the title compound, 5-iodo-2-methyl-3-methylsulfinyl-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 molecular packing (Fig. 2) is stabilized by CH₂—H···π interactions between the hydrogen of 2-methyl group and a benzene ring of benzofuran unit, with a C9—H9B···C_g separation of 2.72 Å (Fig. 2 & Table 1) (C_g is the centroid of C2—C7 benzene ring, symmetry code as in Fig. 2). The molecular packing (Fig. 2) is further stabilized by a I···O halogen bond (Politzer *et al.*, 2007) between the iodine atom and the oxygen of a neighbouring S=O unit, with a I···O2ⁱⁱ distance of 3.139 (2) Å (symmetry code as Fig. 2). In addition, the crystal packing exhibits a sulfinyl-sulfinyl interaction interpreted as similar to a type-II carbonyl-carbonyl interaction (Allen *et al.*, 1998), with S···O2ⁱⁱⁱ and O2···Sⁱⁱⁱ distances of 3.190 (2) Å (symmetry code as Fig. 2).

Experimental

3-Chloroperbenzoic acid (77%, 247 mg, 1.1 mmol) was added in small portions to a stirred solution of 5-iodo-2-methyl-3-methylsulfonyl-1-benzofuran (304 mg, 1.0 mmol) in dichloromethane (30 ml) at 273 K. After stirring 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 (ethyl acetate) to afford the title compound as a colorless solid [yield 80%, m.p. 420–421 K; *R*_f = 0.44 (ethyl acetate)]. Single crystals suitable for X-ray diffraction were prepared by slow evaporation of a dilute solution of the title compound in acetone at room temperature.

Refinement

All H atoms were geometrically located in ideal positions and refined using a riding model, with C—H=0.93 Å for aromatic H atoms and 0.96 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. The highest peak in the difference map is 0.85 Å from I.

Figures

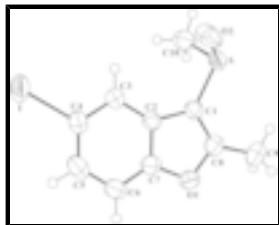


Fig. 1. The molecular structure of the title compound, showing displacement ellipsoids drawn at the 30% probability level.

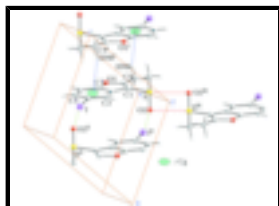


Fig. 2. C—H... π , I...O halogen bond and S...O interaction (dotted lines) in the title compound. Cg denotes ring centroid. [Symmetry code: (i) $-x + 1, -y, -z + 1$; (ii) $-x, -y + 1, -z + 1$; (iii) $-x, -y, -z$.]

5-Iodo-2-methyl-3-methylsulfinyl-1-benzofuran

Crystal data

$C_{10}H_9IO_2S$	$Z = 2$
$M_r = 320.13$	$F_{000} = 308$
Triclinic, $P\bar{1}$	$D_x = 1.938 \text{ Mg m}^{-3}$
Hall symbol: $-P\ 1$	Mo $K\alpha$ radiation
$a = 8.1521(4) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 8.2671(4) \text{ \AA}$	Cell parameters from 8333 reflections
$c = 8.6870(4) \text{ \AA}$	$\theta = 2.5\text{--}28.3^\circ$
$\alpha = 104.585(2)^\circ$	$\mu = 3.08 \text{ mm}^{-1}$
$\beta = 99.920(2)^\circ$	$T = 298(2) \text{ K}$
$\gamma = 97.620(2)^\circ$	Block, silver
$V = 548.52(5) \text{ \AA}^3$	$0.41 \times 0.35 \times 0.20 \text{ mm}$

Data collection

Bruker SMART CCD diffractometer	2695 independent reflections
Radiation source: fine-focus sealed tube	2448 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.017$
Detector resolution: $10.0 \text{ pixels mm}^{-1}$	$\theta_{\text{max}} = 28.4^\circ$
$T = 298(2) \text{ K}$	$\theta_{\text{min}} = 2.5^\circ$
φ and ω scans	$h = -10 \rightarrow 9$
Absorption correction: multi-scan (SADABS; Sheldrick, 2000)	$k = -11 \rightarrow 10$
$T_{\text{min}} = 0.301, T_{\text{max}} = 0.538$	$l = -11 \rightarrow 11$
10576 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.028$	H-atom parameters constrained
$wR(F^2) = 0.066$	$w = 1/[\sigma^2(F_o^2) + (0.0241P)^2 + 0.6885P]$
$S = 1.08$	where $P = (F_o^2 + 2F_c^2)/3$
2695 reflections	$(\Delta/\sigma)_{\max} = <0.001$
128 parameters	$\Delta\rho_{\max} = 1.31 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.73 \text{ e } \text{\AA}^{-3}$
	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 $F^2 > 2\sigma(F^2)$ 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	$U_{\text{iso}}^*/U_{\text{eq}}$
I	0.14093 (3)	0.58796 (3)	0.80417 (3)	0.06018 (9)
S	0.19880 (9)	0.03223 (9)	0.15991 (8)	0.04263 (16)
O1	0.6390 (2)	0.1957 (3)	0.4697 (2)	0.0459 (4)
O2	0.1218 (3)	0.1740 (3)	0.1195 (3)	0.0624 (6)
C1	0.3697 (3)	0.1256 (3)	0.3272 (3)	0.0384 (5)
C2	0.3747 (3)	0.2544 (3)	0.4761 (3)	0.0376 (5)
C3	0.2573 (4)	0.3385 (4)	0.5464 (3)	0.0410 (6)
H3	0.1439	0.3164	0.4936	0.049*
C4	0.3155 (4)	0.4566 (4)	0.6980 (3)	0.0442 (6)
C5	0.4861 (4)	0.4939 (4)	0.7772 (4)	0.0515 (7)
H5	0.5209	0.5748	0.8781	0.062*
C6	0.6037 (4)	0.4119 (4)	0.7075 (4)	0.0501 (7)
H6	0.7177	0.4363	0.7587	0.060*
C7	0.5440 (4)	0.2926 (4)	0.5589 (3)	0.0419 (6)
C8	0.5284 (4)	0.0956 (4)	0.3289 (3)	0.0414 (6)
C9	0.6034 (4)	-0.0215 (4)	0.2141 (4)	0.0519 (7)
H9A	0.5166	-0.0860	0.1213	0.078*
H9B	0.6530	-0.0975	0.2673	0.078*

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H9C	0.6892	0.0430	0.1792	0.078*
C10	0.0620 (4)	-0.0694 (5)	0.2611 (4)	0.0599 (8)
H10A	-0.0414	-0.1285	0.1850	0.090*
H10B	0.0369	0.0146	0.3481	0.090*
H10C	0.1168	-0.1490	0.3047	0.090*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I	0.06932 (16)	0.04801 (13)	0.05745 (14)	0.00644 (10)	0.02787 (11)	-0.00282 (9)
S	0.0476 (4)	0.0447 (4)	0.0317 (3)	0.0149 (3)	0.0052 (3)	0.0028 (3)
O1	0.0366 (10)	0.0573 (12)	0.0465 (11)	0.0108 (9)	0.0089 (8)	0.0184 (9)
O2	0.0724 (16)	0.0553 (13)	0.0499 (12)	0.0221 (11)	-0.0116 (11)	0.0088 (10)
C1	0.0401 (13)	0.0418 (13)	0.0339 (12)	0.0108 (11)	0.0071 (10)	0.0105 (10)
C2	0.0393 (13)	0.0415 (13)	0.0327 (12)	0.0074 (10)	0.0059 (10)	0.0128 (10)
C3	0.0410 (14)	0.0423 (14)	0.0371 (13)	0.0074 (11)	0.0071 (11)	0.0079 (11)
C4	0.0534 (16)	0.0392 (13)	0.0380 (13)	0.0051 (12)	0.0140 (12)	0.0066 (11)
C5	0.0615 (19)	0.0483 (16)	0.0355 (14)	-0.0054 (14)	0.0058 (13)	0.0063 (12)
C6	0.0434 (15)	0.0576 (18)	0.0424 (15)	-0.0034 (13)	0.0010 (12)	0.0138 (13)
C7	0.0391 (14)	0.0480 (15)	0.0406 (14)	0.0052 (11)	0.0088 (11)	0.0171 (12)
C8	0.0447 (15)	0.0448 (14)	0.0406 (14)	0.0122 (11)	0.0124 (11)	0.0182 (12)
C9	0.0546 (18)	0.0551 (17)	0.0577 (18)	0.0214 (14)	0.0259 (14)	0.0216 (15)
C10	0.0468 (17)	0.073 (2)	0.0527 (18)	0.0012 (16)	0.0032 (14)	0.0161 (17)

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

I—O2 ⁱ	3.139 (2)	C3—H3	0.9300
I—C4	2.100 (3)	C4—C5	1.397 (4)
S—O2	1.489 (2)	C5—C6	1.382 (5)
S—O2 ⁱⁱ	3.190 (2)	C5—H5	0.9300
S—C1	1.761 (3)	C6—C7	1.375 (4)
S—C10	1.784 (4)	C6—H6	0.9300
O1—C7	1.379 (4)	C8—C9	1.478 (4)
O1—C8	1.380 (4)	C9—H9A	0.9600
O2—S ⁱⁱ	3.190 (2)	C9—H9B	0.9600
C1—C2	1.444 (4)	C9—H9C	0.9600
C1—C8	1.347 (4)	C10—H10A	0.9600
C2—C3	1.392 (4)	C10—H10B	0.9600
C2—C7	1.396 (4)	C10—H10C	0.9600
C3—C4	1.388 (4)		
O2—S—C1	106.65 (13)	C7—C6—H6	121.6
O2—S—C10	107.45 (17)	C5—C6—H6	121.6
C1—S—C10	98.26 (14)	C6—C7—O1	126.2 (3)
C4—I—O2 ⁱ	164.13 (9)	C6—C7—C2	123.6 (3)
C7—O1—C8	106.4 (2)	O1—C7—C2	110.3 (2)
C8—C1—C2	107.5 (2)	C1—C8—O1	111.0 (2)
C8—C1—S	123.4 (2)	C1—C8—C9	133.2 (3)
C2—C1—S	128.9 (2)	O1—C8—C9	115.8 (3)

C3—C2—C7	119.3 (3)	C8—C9—H9A	109.5
C3—C2—C1	135.8 (3)	C8—C9—H9B	109.5
C7—C2—C1	104.9 (2)	H9A—C9—H9B	109.5
C4—C3—C2	117.6 (3)	C8—C9—H9C	109.5
C4—C3—H3	121.2	H9A—C9—H9C	109.5
C2—C3—H3	121.2	H9B—C9—H9C	109.5
C3—C4—C5	121.9 (3)	S—C10—H10A	109.5
C3—C4—I	118.2 (2)	S—C10—H10B	109.5
C5—C4—I	119.9 (2)	H10A—C10—H10B	109.5
C6—C5—C4	120.8 (3)	S—C10—H10C	109.5
C6—C5—H5	119.6	H10A—C10—H10C	109.5
C4—C5—H5	119.6	H10B—C10—H10C	109.5
C7—C6—C5	116.8 (3)		
O2—S—C1—C8	-126.7 (3)	C5—C6—C7—O1	-179.3 (3)
C10—S—C1—C8	122.2 (3)	C5—C6—C7—C2	1.4 (4)
O2—S—C1—C2	47.1 (3)	C8—O1—C7—C6	-179.3 (3)
C10—S—C1—C2	-64.0 (3)	C8—O1—C7—C2	0.1 (3)
C8—C1—C2—C3	-179.8 (3)	C3—C2—C7—C6	-0.9 (4)
S—C1—C2—C3	5.6 (5)	C1—C2—C7—C6	179.4 (3)
C8—C1—C2—C7	-0.1 (3)	C3—C2—C7—O1	179.7 (2)
S—C1—C2—C7	-174.7 (2)	C1—C2—C7—O1	0.0 (3)
C7—C2—C3—C4	-0.6 (4)	C2—C1—C8—O1	0.2 (3)
C1—C2—C3—C4	179.1 (3)	S—C1—C8—O1	175.18 (19)
C2—C3—C4—C5	1.4 (4)	C2—C1—C8—C9	179.2 (3)
C2—C3—C4—I	178.9 (2)	S—C1—C8—C9	-5.8 (5)
C3—C4—C5—C6	-0.9 (5)	C7—O1—C8—C1	-0.2 (3)
I—C4—C5—C6	-178.4 (2)	C7—O1—C8—C9	-179.4 (2)
C4—C5—C6—C7	-0.5 (5)		

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C9—H9B \cdots Cg ⁱⁱⁱ	0.96	2.72	3.527 (3)	142

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

Fig. 1

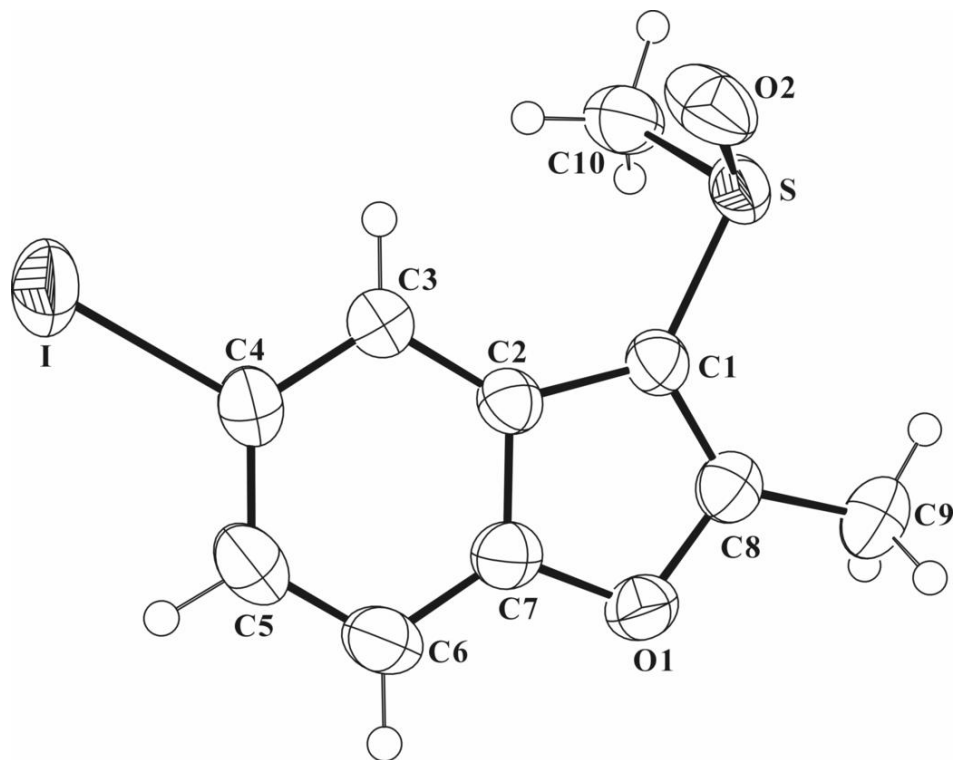


Fig. 2

