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2-(4-Fluorophenyl)-5-iodo-3-isopropylsulfanyl-1-benzofuran

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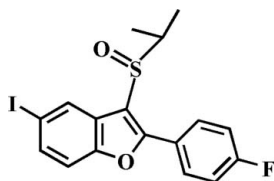
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 Key indicators: single-crystal X-ray study; $T = 180$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.020; wR factor = 0.055; data-to-parameter ratio = 17.6.

In the title compound, $\text{C}_{17}\text{H}_{14}\text{FIO}_2\text{S}$, the 4-fluorophenyl ring makes a dihedral angle of 18.88 (9°) with the mean plane of the benzofuran ring. In the crystal, pairs of intermolecular $\text{I} \cdots \text{O}$ contacts [3.153 (2) Å] link the molecules into inversion dimers.

Related literature

For the pharmacological 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 our previous structural studies of related 3-alkylsulfanyl-2-(4-fluorophenyl)-5-iodo-1-benzofuran derivatives, see: Choi *et al.* (2010*a,b*). For a review of halogen bonding, see: Politzer *et al.* (2007).



Experimental

Crystal data

 $\text{C}_{17}\text{H}_{14}\text{FIO}_2\text{S}$
 $M_r = 428.24$

Triclinic, $P\bar{1}$
 $a = 8.4345$ (1) Å
 $b = 9.5874$ (2) Å
 $c = 10.9324$ (2) Å
 $\alpha = 68.643$ (1°)
 $\beta = 70.618$ (1°)
 $\gamma = 89.241$ (1°)

$V = 770.83$ (2) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 2.23$ mm⁻¹
 $T = 180$ K
 $0.23 \times 0.22 \times 0.12$ mm

Data collection

Bruker SMART APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2009)
 $T_{\min} = 0.594$, $T_{\max} = 0.746$

13711 measured reflections
 3544 independent reflections
 3407 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.028$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.020$
 $wR(F^2) = 0.055$
 $S = 1.14$
 3544 reflections

201 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.52$ e Å⁻³
 $\Delta\rho_{\min} = -0.93$ e Å⁻³

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.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2229).

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2-(4-Fluorophenyl)-5-iodo-3-isopropylsulfinyl-1-benzofuran**Hong Dae Choi, Pil Ja Seo, Byeng Wha Son and Uk Lee****S1. Comment**

Many compounds involving a benzofuran ring have received much attention in view of their potent pharmacological properties such as antifungal, antitumor and antiviral, antimicrobial 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 continuing studies of the substituent effect on the solid state structures of 3-alkylsulfinyl-2-(4-fluorophenyl)-5-iodo-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 0.009 (2) Å from the least-squares plane defined by the nine constituent atoms. The dihedral angle formed by the mean plane of the benzofuran ring and the 4-fluorophenyl ring is 18.88 (9)°. The molecular packing is stabilized by an I⋯O halogen-bonding between the iodine and the oxygen of the S=O unit [I1⋯O2ⁱ = 3.153 (2) Å; C4—I1⋯O2ⁱ = 170.73 (6)°; (i) - x + 1, - y, - z + 2] (Poltzer *et al.*, 2007).

S2. Experimental

77% 3-chloroperoxybenzoic acid (224 mg, 1.0 mmol) was added in small portions to a stirred solution of 2-(4-fluorophenyl)-5-iodo-3-isopropylsulfonyl-1-benzofuran (371 mg, 0.9 mmol) in dichloromethane (40 mL) at 273 K. After being stirred at room temperature for 3h, 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 (hexane-ethyl acetate, 2:1 v/v) to afford the title compound as a colorless solid [yield 76%, m.p. 430–431 K. R_f = 0.65 (hexane-ethyl acetate, 2:1 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.

S3. Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95 Å for aryl, 1.00 Å for methine, and 0.98 Å for methyl H atoms. $U_{iso}(H) = 1.2U_{eq}(C)$ for aryl and methine, and $1.5U_{eq}(C)$ for methyl H atoms.

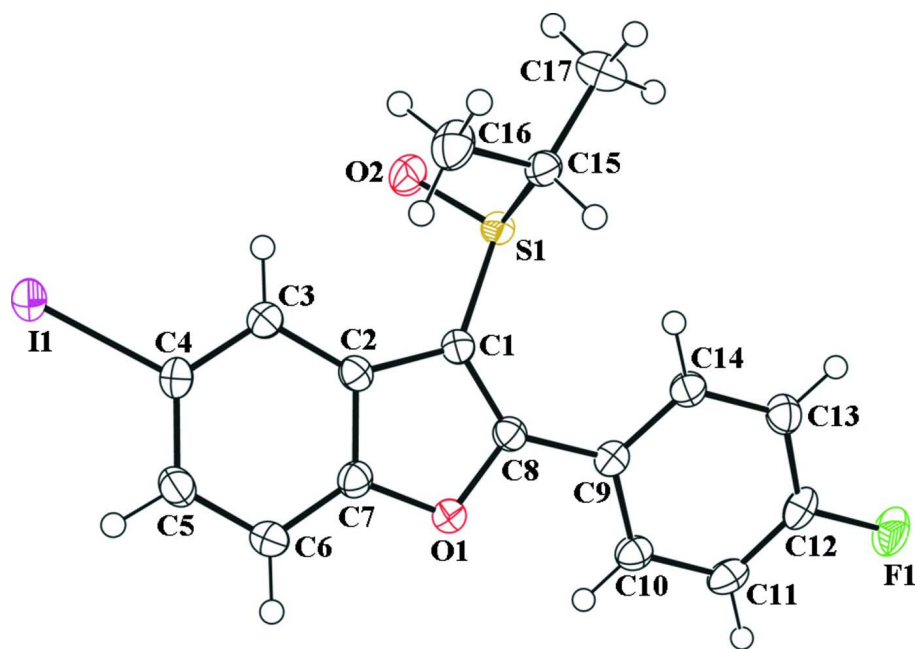


Figure 1

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.

2-(4-Fluorophenyl)-5-iodo-3-isopropylsulfinyl-1-benzofuran

Crystal data

$C_{17}H_{14}FIO_2S$

$M_r = 428.24$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 8.4345$ (1) Å

$b = 9.5874$ (2) Å

$c = 10.9324$ (2) Å

$\alpha = 68.643$ (1)°

$\beta = 70.618$ (1)°

$\gamma = 89.241$ (1)°

$V = 770.83$ (2) Å³

$Z = 2$

$F(000) = 420$

$D_x = 1.845$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9898 reflections

$\theta = 2.3$ – 27.5 °

$\mu = 2.23$ mm⁻¹

$T = 180$ K

Block, colourless

$0.23 \times 0.22 \times 0.12$ mm

Data collection

Bruker SMART APEXII CCD
diffractometer

Radiation source: rotating anode

Graphite multilayer monochromator

Detector resolution: 10.0 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2009)

$T_{\min} = 0.594$, $T_{\max} = 0.746$

13711 measured reflections

3544 independent reflections

3407 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.028$

$\theta_{\max} = 27.5$ °, $\theta_{\min} = 2.1$ °

$h = -10 \rightarrow 10$

$k = -12 \rightarrow 12$

$l = -14 \rightarrow 14$

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.020$ $wR(F^2) = 0.055$ $S = 1.14$

3544 reflections

201 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
map

Hydrogen site location: difference Fourier map

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0298P)^2 + 0.238P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.52 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.93 \text{ e } \text{\AA}^{-3}$ *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>	$U_{\text{iso}}^*/U_{\text{eq}}$
I1	0.417391 (15)	0.151710 (13)	1.131578 (12)	0.02794 (6)
S1	0.35022 (6)	0.16149 (5)	0.56324 (5)	0.02224 (10)
F1	0.08962 (18)	0.68702 (15)	0.06579 (14)	0.0395 (3)
O1	0.27453 (17)	0.55572 (14)	0.60153 (14)	0.0241 (3)
O2	0.46705 (19)	0.07898 (17)	0.63276 (17)	0.0324 (3)
C1	0.3041 (2)	0.3160 (2)	0.61705 (19)	0.0213 (3)
C2	0.3257 (2)	0.3290 (2)	0.7385 (2)	0.0213 (3)
C3	0.3567 (2)	0.2321 (2)	0.8569 (2)	0.0238 (4)
H3	0.3697	0.1293	0.8716	0.029*
C4	0.3677 (2)	0.2918 (2)	0.9521 (2)	0.0240 (4)
C5	0.3511 (3)	0.4434 (2)	0.9322 (2)	0.0279 (4)
H5	0.3611	0.4801	0.9995	0.034*
C6	0.3203 (3)	0.5403 (2)	0.8147 (2)	0.0284 (4)
H6	0.3087	0.6435	0.7991	0.034*
C7	0.3074 (2)	0.4790 (2)	0.7221 (2)	0.0234 (4)
C8	0.2727 (2)	0.4542 (2)	0.5392 (2)	0.0216 (3)
C9	0.2301 (2)	0.5145 (2)	0.4121 (2)	0.0221 (4)
C10	0.2428 (2)	0.6710 (2)	0.3413 (2)	0.0253 (4)
H10	0.2834	0.7374	0.3739	0.030*
C11	0.1966 (3)	0.7288 (2)	0.2242 (2)	0.0290 (4)
H11	0.2063	0.8348	0.1752	0.035*
C12	0.1363 (2)	0.6309 (2)	0.1796 (2)	0.0273 (4)
C13	0.1226 (3)	0.4763 (2)	0.2458 (2)	0.0278 (4)
H13	0.0818	0.4111	0.2120	0.033*
C14	0.1698 (3)	0.4187 (2)	0.3627 (2)	0.0264 (4)

H14	0.1611	0.3125	0.4100	0.032*
C15	0.1418 (2)	0.0465 (2)	0.6546 (2)	0.0245 (4)
H15	0.0542	0.1115	0.6309	0.029*
C16	0.0973 (3)	-0.0158 (3)	0.8118 (2)	0.0350 (5)
H16A	-0.0086	-0.0843	0.8568	0.053*
H16B	0.0833	0.0675	0.8448	0.053*
H16C	0.1883	-0.0709	0.8359	0.053*
C17	0.1496 (3)	-0.0768 (2)	0.5953 (3)	0.0396 (5)
H17A	0.2342	-0.1422	0.6187	0.059*
H17B	0.1811	-0.0299	0.4932	0.059*
H17C	0.0384	-0.1370	0.6362	0.059*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.03365 (9)	0.02957 (8)	0.02477 (9)	0.00348 (5)	-0.01555 (6)	-0.01020 (6)
S1	0.0247 (2)	0.0215 (2)	0.0218 (2)	0.00425 (17)	-0.00912 (18)	-0.00885 (17)
F1	0.0464 (8)	0.0401 (7)	0.0277 (7)	0.0044 (6)	-0.0213 (6)	-0.0007 (5)
O1	0.0285 (7)	0.0208 (6)	0.0257 (7)	0.0037 (5)	-0.0134 (6)	-0.0084 (5)
O2	0.0322 (8)	0.0346 (7)	0.0376 (8)	0.0131 (6)	-0.0195 (7)	-0.0156 (7)
C1	0.0214 (8)	0.0220 (8)	0.0212 (9)	0.0022 (6)	-0.0080 (7)	-0.0084 (7)
C2	0.0190 (8)	0.0236 (8)	0.0227 (9)	0.0022 (6)	-0.0069 (7)	-0.0106 (7)
C3	0.0244 (9)	0.0237 (8)	0.0253 (10)	0.0048 (7)	-0.0106 (7)	-0.0099 (7)
C4	0.0225 (9)	0.0287 (9)	0.0226 (9)	0.0035 (7)	-0.0109 (7)	-0.0094 (8)
C5	0.0298 (10)	0.0309 (10)	0.0305 (11)	0.0048 (8)	-0.0139 (8)	-0.0169 (8)
C6	0.0335 (10)	0.0241 (9)	0.0342 (11)	0.0061 (8)	-0.0163 (9)	-0.0144 (8)
C7	0.0213 (9)	0.0240 (8)	0.0254 (10)	0.0031 (7)	-0.0103 (7)	-0.0084 (7)
C8	0.0202 (8)	0.0219 (8)	0.0226 (9)	0.0013 (6)	-0.0074 (7)	-0.0086 (7)
C9	0.0189 (8)	0.0226 (8)	0.0211 (9)	0.0015 (6)	-0.0056 (7)	-0.0054 (7)
C10	0.0235 (9)	0.0225 (8)	0.0272 (10)	0.0023 (7)	-0.0080 (8)	-0.0072 (8)
C11	0.0286 (10)	0.0212 (8)	0.0300 (11)	0.0039 (7)	-0.0095 (8)	-0.0026 (8)
C12	0.0239 (9)	0.0315 (9)	0.0208 (9)	0.0048 (7)	-0.0093 (7)	-0.0022 (8)
C13	0.0295 (10)	0.0291 (9)	0.0245 (10)	0.0002 (8)	-0.0110 (8)	-0.0083 (8)
C14	0.0320 (10)	0.0206 (8)	0.0247 (10)	0.0003 (7)	-0.0126 (8)	-0.0042 (7)
C15	0.0251 (9)	0.0205 (8)	0.0272 (10)	0.0017 (7)	-0.0118 (8)	-0.0059 (7)
C16	0.0344 (11)	0.0356 (11)	0.0258 (11)	-0.0043 (9)	-0.0104 (9)	-0.0014 (9)
C17	0.0460 (13)	0.0288 (10)	0.0476 (14)	0.0008 (9)	-0.0163 (11)	-0.0185 (10)

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

I1—C4	2.1074 (19)	C9—C14	1.397 (3)
I1—O2 ⁱ	3.1525 (15)	C9—C10	1.402 (2)
S1—O2	1.4876 (15)	C10—C11	1.381 (3)
S1—C1	1.7774 (18)	C10—H10	0.9500
S1—C15	1.841 (2)	C11—C12	1.374 (3)
F1—C12	1.351 (2)	C11—H11	0.9500
O1—C7	1.373 (2)	C12—C13	1.380 (3)
O1—C8	1.380 (2)	C13—C14	1.384 (3)

C1—C8	1.367 (3)	C13—H13	0.9500
C1—C2	1.446 (3)	C14—H14	0.9500
C2—C3	1.396 (3)	C15—C16	1.510 (3)
C2—C7	1.396 (2)	C15—C17	1.532 (3)
C3—C4	1.385 (3)	C15—H15	1.0000
C3—H3	0.9500	C16—H16A	0.9800
C4—C5	1.402 (3)	C16—H16B	0.9800
C5—C6	1.388 (3)	C16—H16C	0.9800
C5—H5	0.9500	C17—H17A	0.9800
C6—C7	1.375 (3)	C17—H17B	0.9800
C6—H6	0.9500	C17—H17C	0.9800
C8—C9	1.460 (3)		
C4—I1—O2 ⁱ	170.73 (6)	C11—C10—H10	119.9
O2—S1—C1	106.47 (8)	C9—C10—H10	119.9
O2—S1—C15	107.12 (9)	C12—C11—C10	119.07 (18)
C1—S1—C15	99.87 (9)	C12—C11—H11	120.5
C7—O1—C8	106.69 (14)	C10—C11—H11	120.5
C8—C1—C2	106.99 (15)	F1—C12—C11	119.27 (17)
C8—C1—S1	126.19 (15)	F1—C12—C13	118.10 (18)
C2—C1—S1	125.82 (13)	C11—C12—C13	122.63 (19)
C3—C2—C7	118.97 (17)	C12—C13—C14	118.15 (18)
C3—C2—C1	135.97 (17)	C12—C13—H13	120.9
C7—C2—C1	105.06 (16)	C14—C13—H13	120.9
C4—C3—C2	117.37 (17)	C13—C14—C9	120.93 (17)
C4—C3—H3	121.3	C13—C14—H14	119.5
C2—C3—H3	121.3	C9—C14—H14	119.5
C3—C4—C5	122.52 (18)	C16—C15—C17	113.13 (17)
C3—C4—I1	119.10 (14)	C16—C15—S1	111.52 (14)
C5—C4—I1	118.36 (14)	C17—C15—S1	106.42 (14)
C6—C5—C4	120.40 (18)	C16—C15—H15	108.5
C6—C5—H5	119.8	C17—C15—H15	108.5
C4—C5—H5	119.8	S1—C15—H15	108.5
C7—C6—C5	116.43 (17)	C15—C16—H16A	109.5
C7—C6—H6	121.8	C15—C16—H16B	109.5
C5—C6—H6	121.8	H16A—C16—H16B	109.5
O1—C7—C6	125.06 (16)	C15—C16—H16C	109.5
O1—C7—C2	110.64 (16)	H16A—C16—H16C	109.5
C6—C7—C2	124.30 (18)	H16B—C16—H16C	109.5
C1—C8—O1	110.61 (16)	C15—C17—H17A	109.5
C1—C8—C9	134.93 (17)	C15—C17—H17B	109.5
O1—C8—C9	114.36 (15)	H17A—C17—H17B	109.5
C14—C9—C10	119.02 (18)	C15—C17—H17C	109.5
C14—C9—C8	121.04 (16)	H17A—C17—H17C	109.5
C10—C9—C8	119.88 (17)	H17B—C17—H17C	109.5
C11—C10—C9	120.20 (18)		
O2—S1—C1—C8	147.63 (17)	S1—C1—C8—O1	-168.30 (13)

C15—S1—C1—C8	-101.09 (18)	C2—C1—C8—C9	-175.53 (19)
O2—S1—C1—C2	-19.38 (18)	S1—C1—C8—C9	15.5 (3)
C15—S1—C1—C2	91.91 (17)	C7—O1—C8—C1	-0.2 (2)
C8—C1—C2—C3	178.6 (2)	C7—O1—C8—C9	176.86 (15)
S1—C1—C2—C3	-12.3 (3)	C1—C8—C9—C14	17.1 (3)
C8—C1—C2—C7	-0.9 (2)	O1—C8—C9—C14	-159.09 (17)
S1—C1—C2—C7	168.15 (14)	C1—C8—C9—C10	-165.7 (2)
C7—C2—C3—C4	0.0 (3)	O1—C8—C9—C10	18.2 (2)
C1—C2—C3—C4	-179.5 (2)	C14—C9—C10—C11	-0.2 (3)
C2—C3—C4—C5	-0.8 (3)	C8—C9—C10—C11	-177.49 (17)
C2—C3—C4—I1	-179.23 (13)	C9—C10—C11—C12	0.8 (3)
C3—C4—C5—C6	0.9 (3)	C10—C11—C12—F1	179.47 (18)
I1—C4—C5—C6	179.27 (15)	C10—C11—C12—C13	-1.1 (3)
C4—C5—C6—C7	0.0 (3)	F1—C12—C13—C14	-179.79 (18)
C8—O1—C7—C6	179.81 (18)	C11—C12—C13—C14	0.8 (3)
C8—O1—C7—C2	-0.40 (19)	C12—C13—C14—C9	-0.1 (3)
C5—C6—C7—O1	178.82 (18)	C10—C9—C14—C13	-0.2 (3)
C5—C6—C7—C2	-0.9 (3)	C8—C9—C14—C13	177.14 (18)
C3—C2—C7—O1	-178.83 (16)	O2—S1—C15—C16	43.28 (16)
C1—C2—C7—O1	0.8 (2)	C1—S1—C15—C16	-67.49 (15)
C3—C2—C7—C6	1.0 (3)	O2—S1—C15—C17	-80.53 (15)
C1—C2—C7—C6	-179.40 (18)	C1—S1—C15—C17	168.70 (14)
C2—C1—C8—O1	0.7 (2)		

Symmetry code: (i) $-x+1, -y, -z+2$.