Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-Fluoro-2-(4-fluorophenyl)-3-methylsulfinyl-1-benzofuran

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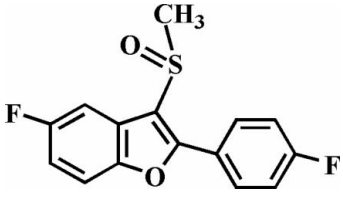
Received 25 September 2009; accepted 28 September 2009

Key indicators: single-crystal X-ray study; T = 172 K; mean σ(C–C) = 0.002 Å; R factor = 0.030; wR factor = 0.084; data-to-parameter ratio = 15.9.

In the title compound, $C_{15}H_{10}F_2O_2S$, the O atom and the methyl group of the methylsulfinyl substituent lie on opposite sides of the plane through the benzofuran fragment. The 4-fluorophenyl ring is rotated out of the benzofuran plane by a dihedral angle of 28.09 (3)°. The crystal structure is stabilized by weak intermolecular C—H···O and C—H···F hydrogen bonds.

Related literature

For the crystal structures of similar 5-fluoro-2-(4-halophenyl)-3-methylsulfinyl-1-benzofuran derivatives, see: Choi *et al.* (2009*a*,*b*). For the biological activity of benzofuran compounds, see: Howlett *et al.* (1999); Twyman & Allsop (1999). For natural products with benzofuran rings, see: Akgul & Anil (2003); Soekamto *et al.* (2003).



Experimental

Crystal data

$C_{15}H_{10}F_2O_2S$ M_r = 292.29
Triclinic, $P\overline{1}$
a = 7.9275 (3) Å
b = 8.2069 (3) Å
c = 10.6822 (4) Å
α = 97.033 (2)°
β = 91.516 (2)°
γ = 113.533 (2)°
V = 630.22 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 172 K
0.44 × 0.23 × 0.12 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.888, T_{max} = 0.968
10883 measured reflections
2900 independent reflections
2661 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.084
S = 1.06
2900 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C14—H14···O2^{i}	0.95	2.54	3.4025 (17)	151
C15—H15C···F2^{ii}	0.98	2.52	3.2235 (16)	129

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

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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2465).

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supporting information

Acta Cryst. (2009). E**65**, o2608 [https://doi.org/10.1107/S1600536809039312]

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S1. Comment

Benzofuran ring systems have received considerable attention in view of a wide range of biological activities (Howlett *et al.*, 1999; Twyman & Allsop, 1999) and these compounds are ubiquitous in nature (Akgul & Anil, 2003; Soekamto *et al.*, 2003). As a part of our ongoing studies of the effect of side chain substituents on the solid state structures of 5-fluoro-2-(4-halophenyl)-3-methylsulfinyl-1-benzofuran analogues (Choi *et al.*, 2009*a,b*), we report the crystal structure of the title compound (Fig. 1).

The benzofuran unit is essentially planar, with a mean deviation of 0.004 (1) Å from the least-squares plane defined by the nine constituent atoms. The dihedral angle formed by the plane of the benzofuran ring and the 4-fluorophenyl ring is 28.09 (3)°. The crystal packing (Fig. 2) is stabilized by weak non-classical intermolecular C–H···O and C–H···F hydrogen bonds; the first between the 4-fluorophenyl H atom and the oxygen of the S═O unit, with a C14–H14···O2^{i}, the second between the methyl H atom and the fluorine of the 4-fluorophenyl ring, with a C15–H15C···F2^{ii}, respectively (Table 1).

S2. Experimental

77% 3-Chloroperoxybenzoic acid (359 mg, 1.6 mmol) was added in small portions to a stirred solution of 5-fluoro-2-(4-fluorophenyl)-3-methylsulfanyl-1-benzofuran (414 mg, 1.5 mmol) in dichloromethane (30 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 in vacuum. The residue was purified by column chromatography (hexane-ethyl acetate, 1:1 v/v) to afford the title compound as a colorless solid [yield 80%, m.p. 437-438 K; Rf = 0.54 (hexane-ethyl acetate, 1:1 v/v)]. Single crystals suitable for X-ray diffraction were prepared by slow evaporation of a solution of the title compound in chloroforrm at room temperature.

S3. Refinement

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

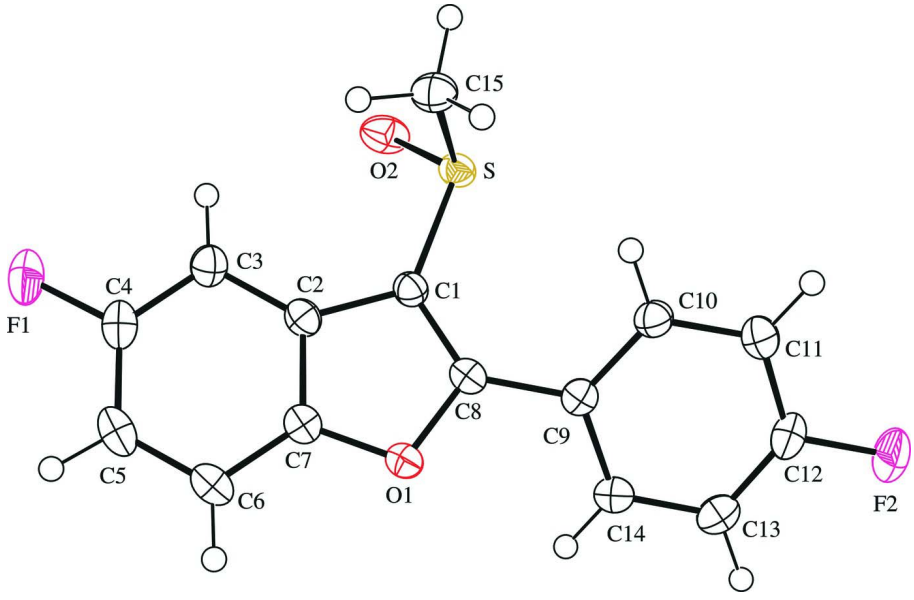

Figure 1

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.

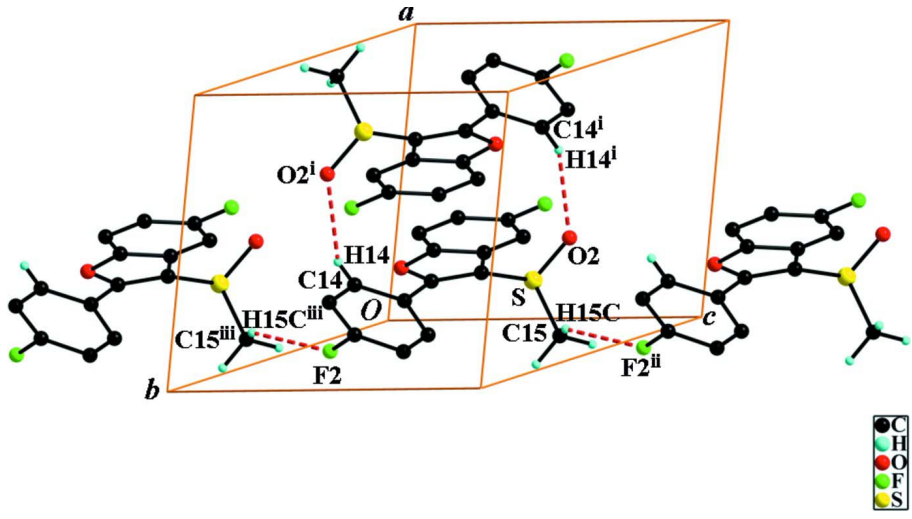

Figure 2

C–H···O and C–H···F hydrogen bonds (dotted lines) in the crystal structure of the title compound. [Symmetry codes: (i) -*x* + 1, - *y* + 1, - *z* + 1; (ii) *x*, *y*, *z* + 1 (iii) *x*, *y*, *z* - 1.]

5-Fluoro-2-(4-fluorophenyl)-3-methylsulfinyl-1-benzofuran

Crystal data

$C_{15}H_{10}F_2O_2S$
M_r = 292.29
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 7.9275 (3) Å
b = 8.2069 (3) Å
c = 10.6822 (4) Å
α = 97.033 (2)°
β = 91.516 (2)°
γ = 113.533 (2)°
V = 630.22 (4) Å^3
Z = 2

$F(000) = 300$
$D_x = 1.540$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8044 reflections
$\theta = 2.7$–$27.5°$
$\mu = 0.28$ mm^{-1}
$T = 172$ K
Block, colorless
0.44 × 0.23 × 0.12 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Radiation source: Rotating Anode
HELIOS monochromator
Detector resolution: 10.0 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*APEX2*; Bruker, 2009)
$T_{min} = 0.888$, $T_{max} = 0.968$
10883 measured reflections
2900 independent reflections
2661 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.9°$
$h = -10 \rightarrow 10$
$k = -10 \rightarrow 10$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.084$
$S = 1.06$
2900 reflections
182 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0414P)^2 + 0.2389P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S	0.17610 (4)	0.19867 (4)	0.58632 (3)	0.02317 (10)
F1	0.56623 (14)	0.77195 (13)	0.99398 (8)	0.0443 (2)
F2	−0.04941 (15)	0.22620 (14)	−0.02234 (8)	0.0495 (3)
O1	0.33749 (13)	0.69196 (11)	0.49937 (8)	0.0246 (2)
O2	0.31172 (14)	0.17318 (13)	0.67238 (10)	0.0348 (2)
C1	0.25777 (17)	0.43064 (16)	0.57768 (11)	0.0217 (2)
C2	0.35892 (17)	0.57414 (16)	0.67846 (12)	0.0230 (2)
C3	0.41294 (19)	0.58468 (18)	0.80585 (12)	0.0272 (3)
H3	0.3843	0.4807	0.8459	0.033*
C4	0.5103 (2)	0.7554 (2)	0.86950 (13)	0.0312 (3)
C5	0.5563 (2)	0.91200 (19)	0.81591 (14)	0.0327 (3)

H5	0.6239	1.0255	0.8656	0.039*
C6	0.50286 (19)	0.90178 (18)	0.68960 (14)	0.0291 (3)
H6	0.5316	1.0062	0.6500	0.035*
C7	0.40540 (17)	0.73123 (17)	0.62445 (12)	0.0239 (3)
C8	0.24884 (17)	0.50769 (16)	0.47293 (12)	0.0222 (2)
C9	0.16701 (17)	0.43530 (17)	0.34357 (11)	0.0223 (2)
C10	0.01310 (18)	0.27185 (18)	0.31677 (12)	0.0258 (3)
H10	−0.0425	0.2097	0.3843	0.031*
C11	−0.05978 (19)	0.19894 (19)	0.19358 (13)	0.0299 (3)
H11	−0.1622	0.0861	0.1751	0.036*
C12	0.0214 (2)	0.2958 (2)	0.09877 (12)	0.0316 (3)
C13	0.1700 (2)	0.45994 (19)	0.12075 (13)	0.0311 (3)
H13	0.2203	0.5238	0.0528	0.037*
C14	0.24436 (19)	0.52965 (17)	0.24403 (12)	0.0259 (3)
H14	0.3481	0.6417	0.2612	0.031*
C15	−0.01622 (19)	0.18188 (19)	0.67642 (13)	0.0295 (3)
H15A	−0.0741	0.0608	0.6994	0.044*
H15B	−0.1065	0.2056	0.6257	0.044*
H15C	0.0268	0.2701	0.7535	0.044*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.02566 (18)	0.01834 (16)	0.02506 (16)	0.00839 (13)	0.00392 (12)	0.00302 (11)
F1	0.0527 (6)	0.0458 (5)	0.0260 (4)	0.0155 (5)	−0.0092 (4)	−0.0068 (4)
F2	0.0558 (6)	0.0565 (6)	0.0222 (4)	0.0102 (5)	−0.0081 (4)	0.0014 (4)
O1	0.0261 (5)	0.0193 (4)	0.0262 (4)	0.0072 (4)	0.0005 (3)	0.0036 (3)
O2	0.0290 (5)	0.0301 (5)	0.0493 (6)	0.0136 (4)	0.0013 (4)	0.0148 (4)
C1	0.0229 (6)	0.0194 (6)	0.0219 (6)	0.0081 (5)	0.0023 (5)	0.0017 (4)
C2	0.0217 (6)	0.0214 (6)	0.0257 (6)	0.0093 (5)	0.0023 (5)	0.0011 (5)
C3	0.0291 (7)	0.0281 (6)	0.0249 (6)	0.0129 (6)	0.0008 (5)	0.0010 (5)
C4	0.0305 (7)	0.0354 (7)	0.0252 (6)	0.0135 (6)	−0.0028 (5)	−0.0041 (5)
C5	0.0275 (7)	0.0262 (7)	0.0376 (7)	0.0078 (6)	−0.0024 (6)	−0.0076 (5)
C6	0.0251 (7)	0.0210 (6)	0.0381 (7)	0.0071 (5)	0.0009 (5)	0.0007 (5)
C7	0.0220 (6)	0.0234 (6)	0.0263 (6)	0.0097 (5)	0.0014 (5)	0.0018 (5)
C8	0.0205 (6)	0.0194 (6)	0.0257 (6)	0.0073 (5)	0.0028 (5)	0.0023 (4)
C9	0.0225 (6)	0.0235 (6)	0.0228 (6)	0.0113 (5)	0.0021 (5)	0.0033 (5)
C10	0.0241 (6)	0.0280 (6)	0.0241 (6)	0.0085 (5)	0.0028 (5)	0.0065 (5)
C11	0.0252 (7)	0.0304 (7)	0.0297 (7)	0.0074 (6)	−0.0026 (5)	0.0024 (5)
C12	0.0333 (7)	0.0398 (8)	0.0208 (6)	0.0152 (6)	−0.0032 (5)	0.0016 (5)
C13	0.0349 (7)	0.0351 (7)	0.0251 (6)	0.0145 (6)	0.0061 (5)	0.0095 (5)
C14	0.0266 (6)	0.0241 (6)	0.0273 (6)	0.0100 (5)	0.0041 (5)	0.0058 (5)
C15	0.0284 (7)	0.0295 (7)	0.0318 (7)	0.0118 (6)	0.0088 (5)	0.0077 (5)

Geometric parameters (Å, º)

S—O2	1.4897 (10)	C6—C7	1.3820 (18)
S—C1	1.7646 (12)	C6—H6	0.9500

S—C15	1.7922 (13)	C8—C9	1.4584 (17)
F1—C4	1.3636 (15)	C9—C10	1.3966 (18)
F2—C12	1.3565 (15)	C9—C14	1.4011 (17)
O1—C7	1.3773 (15)	C10—C11	1.3837 (18)
O1—C8	1.3773 (15)	C10—H10	0.9500
C1—C8	1.3645 (17)	C11—C12	1.377 (2)
C1—C2	1.4417 (17)	C11—H11	0.9500
C2—C7	1.3946 (17)	C12—C13	1.378 (2)
C2—C3	1.3977 (17)	C13—C14	1.3835 (19)
C3—C4	1.3769 (19)	C13—H13	0.9500
C3—H3	0.9500	C14—H14	0.9500
C4—C5	1.389 (2)	C15—H15A	0.9800
C5—C6	1.386 (2)	C15—H15B	0.9800
C5—H5	0.9500	C15—H15C	0.9800

O2—S—C1	107.26 (6)	C1—C8—C9	133.40 (12)
O2—S—C15	106.17 (6)	O1—C8—C9	115.96 (10)
C1—S—C15	97.08 (6)	C10—C9—C14	119.18 (12)
C7—O1—C8	106.55 (9)	C10—C9—C8	121.02 (11)
C8—C1—C2	107.21 (11)	C14—C9—C8	119.79 (12)
C8—C1—S	126.69 (10)	C11—C10—C9	121.14 (12)
C2—C1—S	125.96 (9)	C11—C10—H10	119.4
C7—C2—C3	119.65 (12)	C9—C10—H10	119.4
C7—C2—C1	105.04 (11)	C12—C11—C10	117.62 (13)
C3—C2—C1	135.31 (12)	C12—C11—H11	121.2
C4—C3—C2	115.71 (13)	C10—C11—H11	121.2
C4—C3—H3	122.1	F2—C12—C11	118.17 (13)
C2—C3—H3	122.1	F2—C12—C13	118.46 (12)
F1—C4—C3	117.70 (13)	C11—C12—C13	123.37 (13)
F1—C4—C5	117.60 (12)	C12—C13—C14	118.51 (12)
C3—C4—C5	124.70 (13)	C12—C13—H13	120.7
C6—C5—C4	119.67 (13)	C14—C13—H13	120.7
C6—C5—H5	120.2	C13—C14—C9	120.14 (12)
C4—C5—H5	120.2	C13—C14—H14	119.9
C7—C6—C5	116.25 (13)	C9—C14—H14	119.9
C7—C6—H6	121.9	S—C15—H15A	109.5
C5—C6—H6	121.9	S—C15—H15B	109.5
O1—C7—C6	125.41 (12)	H15A—C15—H15B	109.5
O1—C7—C2	110.56 (11)	S—C15—H15C	109.5
C6—C7—C2	124.02 (12)	H15A—C15—H15C	109.5
C1—C8—O1	110.64 (11)	H15B—C15—H15C	109.5

O2—S—C1—C8	141.63 (11)	C1—C2—C7—C6	179.46 (12)
C15—S—C1—C8	−108.95 (12)	C2—C1—C8—O1	−0.09 (14)
O2—S—C1—C2	−33.41 (12)	S—C1—C8—O1	−175.89 (9)
C15—S—C1—C2	76.01 (12)	C2—C1—C8—C9	179.81 (12)
C8—C1—C2—C7	−0.38 (13)	S—C1—C8—C9	4.0 (2)
S—C1—C2—C7	175.46 (9)	C7—O1—C8—C1	0.53 (13)

C8—C1—C2—C3	179.48 (14)	C7—O1—C8—C9	−179.39 (10)
S—C1—C2—C3	−4.7 (2)	C1—C8—C9—C10	28.3 (2)
C7—C2—C3—C4	0.23 (18)	O1—C8—C9—C10	−151.79 (11)
C1—C2—C3—C4	−179.61 (13)	C1—C8—C9—C14	−151.07 (14)
C2—C3—C4—F1	−179.84 (11)	O1—C8—C9—C14	28.83 (16)
C2—C3—C4—C5	0.0 (2)	C14—C9—C10—C11	2.31 (19)
F1—C4—C5—C6	179.82 (12)	C8—C9—C10—C11	−177.08 (12)
C3—C4—C5—C6	0.0 (2)	C9—C10—C11—C12	−1.9 (2)
C4—C5—C6—C7	−0.2 (2)	C10—C11—C12—F2	−179.66 (12)
C8—O1—C7—C6	−179.50 (12)	C10—C11—C12—C13	0.0 (2)
C8—O1—C7—C2	−0.78 (13)	F2—C12—C13—C14	−178.91 (12)
C5—C6—C7—O1	178.94 (12)	C11—C12—C13—C14	1.4 (2)
C5—C6—C7—C2	0.4 (2)	C12—C13—C14—C9	−1.0 (2)
C3—C2—C7—O1	−179.17 (11)	C10—C9—C14—C13	−0.82 (19)
C1—C2—C7—O1	0.72 (14)	C8—C9—C14—C13	178.58 (11)
C3—C2—C7—C6	−0.4 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14···O2^{i}	0.95	2.54	3.4025 (17)	151
C15—H15*C*···F2^{ii}	0.98	2.52	3.2235 (16)	129

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