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3-(4-Fluorophenylsulfinyl)-2,5-dimethyl-1-benzofuran

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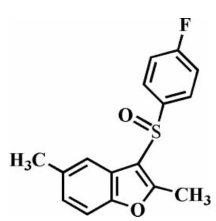
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.035; wR factor = 0.099; data-to-parameter ratio = 17.0.

In the title compound, $C_{16}H_{13}FO_2S$, the O atom and the 4-fluorophenyl group of the 4-fluorophenylsulfinyl substituent are located on opposite sides of the plane through the benzofuran fragment; the 4-fluorophenyl ring is nearly perpendicular to this plane, making a dihedral angle of 87.41 (3). The crystal structure exhibits a weak intermolecular C—H···O hydrogen bond.

Related literature

For the crystal structures of similar 2-methyl-3-phenylsulfinyl-1-benzofuran derivatives, see: Choi *et al.* (2007, 2008*a*,*b*). 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).



Experimental

Crystal data

$C_{16}H_{13}FO_2S$
M_r = 288.32
Monoclinic, $P2_1/c$
a = 11.3951 (5) Å
b = 6.1223 (3) Å
c = 19.6899 (9) Å
β = 100.155 (2)°
V = 1352.13 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 173 K
0.30 × 0.30 × 0.21 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.645, T_{max} = 0.746
22996 measured reflections
3115 independent reflections
2830 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.099
S = 1.07
3115 reflections
183 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10B···O2^{i}	0.98	2.62	3.554 (2)	159

Symmetry code: (i) $-x+2, y+\frac{1}{2}, -z+\frac{3}{2}$.

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: FK2012).

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supplementary materials

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3-(4-Fluorophenylsulfinyl)-2,5-dimethyl-1-benzofuran

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Comment

Molecules containing benzofuran skeleton show various pharmacological activities such as antifungal (Aslam *et al.*, 2006), antitumor and antiviral (Galal *et al.*, 2009), antimicrobial (Khan *et al.*, 2005) properties, and these compounds are widely occurring 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 2-methyl-3-phenylsulfinyl-1-benzofuran analogues (Choi *et al.*, 2007, 2008*a,b*), we report the crystal structure of the title compound (Fig. 1).

The benzofuran unit is essentially planar, with a mean deviation of 0.009 (1) ° from the least-squares plane defined by the nine constituent atoms. The 4-fluorophenyl ring is almost perpendicular to the plane of the benzofuran fragment [87.41 (3)°] and is tilted slightly towards it. The crystal packing (Fig. 2) is stabilized by a weak intermolecular C—H···O hydrogen bond between the methyl H atom and the oxygen of the S═O unit, with a C10—H10B···O2^{i} (Table 1).

Experimental

77% 3-Chloroperoxybenzoic acid (291 mg, 1.3 mmol) was added in small portions to a stirred solution of 3-(4-fluorophenyl-sulfanyl)-2,5-dimethyl-1-benzofuran (326 mg, 1.2 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. 420-421 K; R_f = 0.69 (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 ethyl acetate at room temperature.

Refinement

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

Figures

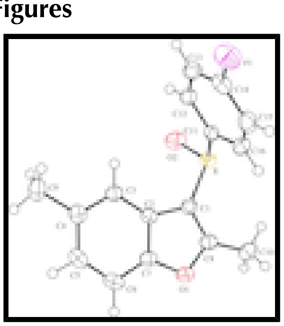

Fig. 1. The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50 % probability level. H atoms are presented as a small spheres of arbitrary radius.

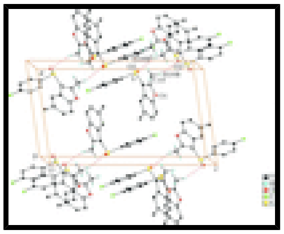

Fig. 2. C–H···O interaction (dotted lines) in the crystal structure of the title compound. [Symmetry codes: (i) - *x* + 2, *y* + 1/2, - *z* + 3/2; (ii) - *x* + 2, *y* - 1/2, - *z* + 3/2.]

3-(4-Fluorophenylsulfinyl)-2,5-dimethyl-1-benzofuran

Crystal data

$C_{16}H_{13}FO_2S$	$F(000) = 600$
$M_r = 288.32$	$D_x = 1.416$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 9959 reflections
$a = 11.3951$ (5) Å	$\theta = 2.5$–$27.6°$
$b = 6.1223$ (3) Å	$\mu = 0.25$ mm^{-1}
$c = 19.6899$ (9) Å	$T = 173$ K
$\beta = 100.155$ (2)°	Block, colourless
$V = 1352.13$ (11) Å^3	0.30 × 0.30 × 0.21 mm
$Z = 4$	

Data collection

Bruker SMART APEXII CCD diffractometer	3115 independent reflections
Radiation source: Rotating Anode	2830 reflections with $I > 2\sigma(I)$
Bruker HELIOS graded multilayer optics	$R_{int} = 0.032$
Detector resolution: 10.0 pixels mm^{-1}	$\theta_{max} = 27.6°$, $\theta_{min} = 1.8°$
φ and ω scans	$h = -14 \rightarrow 14$
Absorption correction: multi-scan (SADABS; Bruker, 2009)	$k = -7 \rightarrow 7$
$T_{min} = 0.645$, $T_{max} = 0.746$	$l = -25 \rightarrow 25$
22996 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.035$	Hydrogen site location: difference Fourier map
$wR(F^2) = 0.099$	H-atom parameters constrained
$S = 1.07$	$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.5136P]$ where $P = (F_o^2 + 2F_c^2)/3$
3115 reflections	$(\Delta/\sigma)_{max} < 0.001$
183 parameters	$\Delta\rho_{max} = 0.30$ e Å^{-3}

0 restraints	$\Delta\rho_{min}$ = −0.31 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 > 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}
S	0.93298 (3)	0.31885 (6)	0.605178 (18)	0.03074 (12)
F1	0.72777 (10)	0.51180 (19)	0.31715 (5)	0.0556 (3)
O1	0.68994 (9)	0.46191 (17)	0.71689 (5)	0.0346 (2)
O2	0.96804 (10)	0.08380 (18)	0.60734 (6)	0.0410 (3)
C1	0.80592 (12)	0.3425 (2)	0.64361 (7)	0.0277 (3)
C2	0.70234 (11)	0.2027 (2)	0.63519 (7)	0.0262 (3)
C3	0.66200 (12)	0.0220 (2)	0.59513 (7)	0.0299 (3)
H3	0.7071	−0.0342	0.5628	0.036*
C4	0.55514 (12)	−0.0758 (2)	0.60278 (7)	0.0320 (3)
C5	0.48975 (13)	0.0120 (3)	0.65052 (7)	0.0358 (3)
H5	0.4167	−0.0556	0.6554	0.043*
C6	0.52732 (13)	0.1926 (3)	0.69067 (8)	0.0369 (3)
H6	0.4820	0.2508	0.7225	0.044*
C7	0.63433 (12)	0.2835 (2)	0.68184 (7)	0.0291 (3)
C8	0.79456 (12)	0.4921 (2)	0.69286 (7)	0.0310 (3)
C9	0.51060 (15)	−0.2747 (3)	0.56091 (9)	0.0433 (4)
H9A	0.5564	−0.2937	0.5236	0.065*
H9B	0.4260	−0.2558	0.5412	0.065*
H9C	0.5204	−0.4041	0.5907	0.065*
C10	0.87069 (16)	0.6753 (3)	0.72348 (8)	0.0419 (4)
H10A	0.9421	0.6830	0.7022	0.063*
H10B	0.8942	0.6518	0.7733	0.063*
H10C	0.8262	0.8126	0.7153	0.063*
C11	0.86493 (11)	0.3744 (2)	0.51764 (7)	0.0266 (3)
C12	0.86759 (12)	0.2162 (2)	0.46786 (7)	0.0305 (3)
H12	0.9008	0.0765	0.4804	0.037*
C13	0.82143 (13)	0.2629 (3)	0.39942 (8)	0.0355 (3)
H13	0.8227	0.1568	0.3644	0.043*
C14	0.77397 (13)	0.4659 (3)	0.38377 (8)	0.0364 (3)
C15	0.77240 (14)	0.6277 (3)	0.43241 (9)	0.0391 (3)
H15	0.7392	0.7672	0.4196	0.047*

C16	0.82041 (13)	0.5810 (2)	0.50024 (8)	0.0348 (3)
H16	0.8228	0.6901	0.5348	0.042*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.02162 (18)	0.0348 (2)	0.0361 (2)	0.00162 (12)	0.00577 (13)	−0.00400 (13)
F1	0.0606 (6)	0.0669 (7)	0.0382 (5)	0.0008 (5)	0.0062 (5)	0.0120 (5)
O1	0.0392 (5)	0.0384 (5)	0.0284 (5)	−0.0003 (4)	0.0118 (4)	−0.0062 (4)
O2	0.0384 (6)	0.0412 (6)	0.0443 (6)	0.0170 (5)	0.0095 (5)	0.0035 (5)
C1	0.0259 (6)	0.0283 (6)	0.0291 (6)	0.0017 (5)	0.0055 (5)	−0.0020 (5)
C2	0.0242 (6)	0.0276 (6)	0.0271 (6)	0.0043 (5)	0.0051 (5)	0.0031 (5)
C3	0.0277 (6)	0.0284 (6)	0.0341 (7)	0.0034 (5)	0.0072 (5)	−0.0016 (5)
C4	0.0297 (7)	0.0301 (7)	0.0348 (7)	0.0004 (5)	0.0021 (5)	0.0047 (5)
C5	0.0285 (7)	0.0438 (8)	0.0358 (7)	−0.0036 (6)	0.0076 (6)	0.0081 (6)
C6	0.0342 (8)	0.0477 (9)	0.0319 (7)	0.0024 (6)	0.0140 (6)	0.0021 (6)
C7	0.0319 (7)	0.0316 (7)	0.0245 (6)	0.0029 (5)	0.0064 (5)	0.0007 (5)
C8	0.0327 (7)	0.0332 (7)	0.0270 (6)	0.0017 (5)	0.0045 (5)	−0.0007 (5)
C9	0.0394 (8)	0.0358 (8)	0.0532 (9)	−0.0082 (6)	0.0044 (7)	−0.0019 (7)
C10	0.0495 (9)	0.0397 (8)	0.0359 (8)	−0.0069 (7)	0.0057 (7)	−0.0108 (6)
C11	0.0204 (6)	0.0265 (6)	0.0350 (7)	−0.0017 (5)	0.0106 (5)	−0.0012 (5)
C12	0.0256 (6)	0.0258 (6)	0.0414 (7)	0.0004 (5)	0.0090 (5)	−0.0041 (5)
C13	0.0312 (7)	0.0388 (7)	0.0381 (8)	−0.0038 (6)	0.0110 (6)	−0.0091 (6)
C14	0.0309 (7)	0.0432 (8)	0.0364 (7)	−0.0050 (6)	0.0097 (6)	0.0058 (6)
C15	0.0397 (8)	0.0295 (7)	0.0497 (9)	0.0032 (6)	0.0125 (7)	0.0077 (6)
C16	0.0362 (7)	0.0269 (7)	0.0436 (8)	0.0026 (5)	0.0135 (6)	−0.0036 (6)

Geometric parameters (Å, °)

S—O2	1.4921 (11)	C8—C10	1.480 (2)
S—C1	1.7540 (13)	C9—H9A	0.9800
S—C11	1.7935 (14)	C9—H9B	0.9800
F1—C14	1.3540 (18)	C9—H9C	0.9800
O1—C8	1.3705 (17)	C10—H10A	0.9800
O1—C7	1.3846 (17)	C10—H10B	0.9800
C1—C8	1.3567 (18)	C10—H10C	0.9800
C1—C2	1.4434 (18)	C11—C12	1.3824 (19)
C2—C3	1.3892 (19)	C11—C16	1.3830 (19)
C2—C7	1.3927 (18)	C12—C13	1.387 (2)
C3—C4	1.3886 (19)	C12—H12	0.9500
C3—H3	0.9500	C13—C14	1.369 (2)
C4—C5	1.405 (2)	C13—H13	0.9500
C4—C9	1.508 (2)	C14—C15	1.380 (2)
C5—C6	1.383 (2)	C15—C16	1.381 (2)
C5—H5	0.9500	C15—H15	0.9500
C6—C7	1.379 (2)	C16—H16	0.9500
C6—H6	0.9500		
O2—S—C1	107.72 (7)	C4—C9—H9B	109.5

O2—S—C11	106.18 (6)	H9A—C9—H9B	109.5
C1—S—C11	98.57 (6)	C4—C9—H9C	109.5
C8—O1—C7	106.49 (10)	H9A—C9—H9C	109.5
C8—C1—C2	107.65 (12)	H9B—C9—H9C	109.5
C8—C1—S	123.65 (11)	C8—C10—H10A	109.5
C2—C1—S	128.47 (10)	C8—C10—H10B	109.5
C3—C2—C7	119.36 (12)	H10A—C10—H10B	109.5
C3—C2—C1	136.00 (12)	C8—C10—H10C	109.5
C7—C2—C1	104.64 (12)	H10A—C10—H10C	109.5
C4—C3—C2	119.37 (12)	H10B—C10—H10C	109.5
C4—C3—H3	120.3	C12—C11—C16	121.21 (13)
C2—C3—H3	120.3	C12—C11—S	119.25 (10)
C3—C4—C5	119.09 (13)	C16—C11—S	119.29 (11)
C3—C4—C9	120.40 (13)	C11—C12—C13	119.50 (13)
C5—C4—C9	120.50 (13)	C11—C12—H12	120.2
C6—C5—C4	122.76 (13)	C13—C12—H12	120.2
C6—C5—H5	118.6	C14—C13—C12	118.22 (13)
C4—C5—H5	118.6	C14—C13—H13	120.9
C7—C6—C5	116.24 (13)	C12—C13—H13	120.9
C7—C6—H6	121.9	F1—C14—C13	118.51 (14)
C5—C6—H6	121.9	F1—C14—C15	118.21 (14)
C6—C7—O1	126.34 (12)	C13—C14—C15	123.27 (14)
C6—C7—C2	123.18 (13)	C14—C15—C16	118.09 (14)
O1—C7—C2	110.48 (12)	C14—C15—H15	121.0
C1—C8—O1	110.74 (12)	C16—C15—H15	121.0
C1—C8—C10	132.86 (14)	C15—C16—C11	119.64 (14)
O1—C8—C10	116.40 (12)	C15—C16—H16	120.2
C4—C9—H9A	109.5	C11—C16—H16	120.2

O2—S—C1—C8	131.07 (12)	C1—C2—C7—O1	0.42 (14)
C11—S—C1—C8	−118.80 (12)	C2—C1—C8—O1	−0.67 (16)
O2—S—C1—C2	−42.67 (14)	S—C1—C8—O1	−175.53 (9)
C11—S—C1—C2	67.46 (13)	C2—C1—C8—C10	−179.96 (15)
C8—C1—C2—C3	−178.80 (15)	S—C1—C8—C10	5.2 (2)
S—C1—C2—C3	−4.3 (2)	C7—O1—C8—C1	0.92 (15)
C8—C1—C2—C7	0.15 (15)	C7—O1—C8—C10	−179.66 (12)
S—C1—C2—C7	174.69 (10)	O2—S—C11—C12	−7.30 (12)
C7—C2—C3—C4	−0.5 (2)	C1—S—C11—C12	−118.67 (11)
C1—C2—C3—C4	178.31 (14)	O2—S—C11—C16	178.43 (11)
C2—C3—C4—C5	0.6 (2)	C1—S—C11—C16	67.07 (12)
C2—C3—C4—C9	−178.81 (13)	C16—C11—C12—C13	−2.0 (2)
C3—C4—C5—C6	−0.1 (2)	S—C11—C12—C13	−176.18 (10)
C9—C4—C5—C6	179.28 (14)	C11—C12—C13—C14	−0.3 (2)
C4—C5—C6—C7	−0.4 (2)	C12—C13—C14—F1	−179.40 (12)
C5—C6—C7—O1	−179.06 (13)	C12—C13—C14—C15	1.6 (2)
C5—C6—C7—C2	0.5 (2)	F1—C14—C15—C16	−179.50 (13)
C8—O1—C7—C6	178.76 (14)	C13—C14—C15—C16	−0.5 (2)
C8—O1—C7—C2	−0.82 (15)	C14—C15—C16—C11	−1.9 (2)
C3—C2—C7—C6	0.0 (2)	C12—C11—C16—C15	3.1 (2)
C1—C2—C7—C6	−179.18 (13)	S—C11—C16—C15	177.30 (11)

C3—C2—C7—O1	179.58 (11)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10B···O2^{i}	0.98	2.62	3.554 (2)	159

Symmetry codes: (i) $-x+2$, $y+1/2$, $-z+3/2$.

Fig. 1

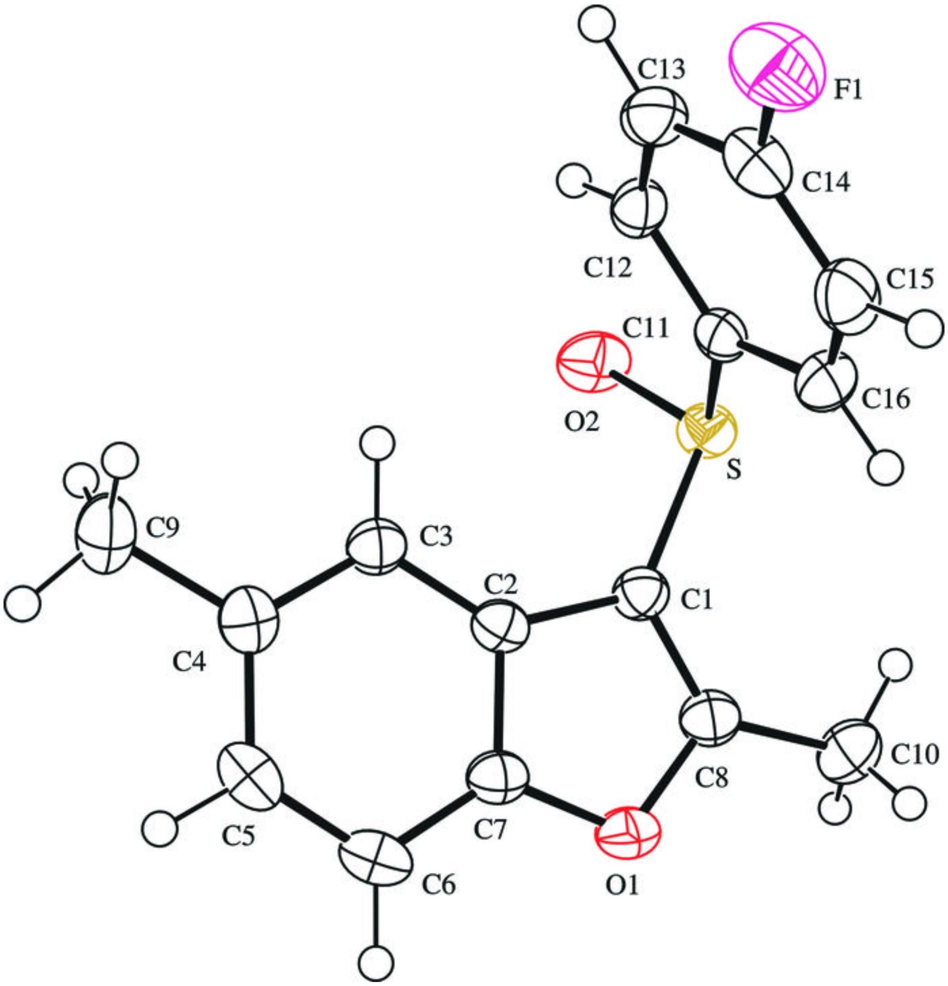

Fig. 2