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

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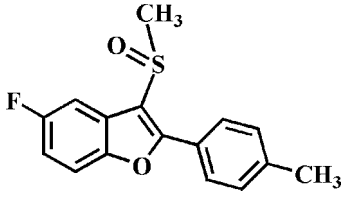
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.034; wR factor = 0.091; data-to-parameter ratio = 18.1.

In the title compound, $C_{16}H_{13}FO_2S$, the 4-methylphenyl ring makes a dihedral angle of 29.53 (4)° with the mean plane of the benzofuran fragment [r.m.s. deviation = 0.004 (1) Å]. In the crystal, molecules are linked by pairs of weak C—H···O hydrogen bonds, forming inversion dimers that stack along the a axis.

Related literature

For background information and the crystal structures of related compounds, see: Choi *et al.* (2009*a*,*b*).



Experimental

Crystal data

$C_{16}H_{13}FO_2S$
M_r = 288.32
Triclinic, $P\overline{1}$
a = 8.0407 (2) Å
b = 8.0838 (2) Å
c = 11.3517 (2) Å
α = 80.126 (1)°
β = 85.091 (1)°
γ = 66.773 (1)°
V = 667.88 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 173 K
0.53 × 0.40 × 0.24 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.701, T_{max} = 0.746
12234 measured reflections
3307 independent reflections
3095 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.091
S = 1.09
3307 reflections
183 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 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	3.4138 (16)	154

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

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2522).

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Choi, H. D., Seo, P. J., Son, B. W. & Lee, U. (2009*b*). *Acta Cryst.* E**65**, o2115.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
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supporting information

Acta Cryst. (2012). E**68**, o3338 [doi:10.1107/S1600536812044844]

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

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S1. Comment

As a part of our ongoing study of 5-fluoro-3-methylsulfinyl-1-benzofuran derivatives with various substituents in the 2-position, such as 4-bromophenyl (Choi *et al.*, 2009*a*) or 4-iodophenyl (Choi *et al.*, 2009*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 0.004 (1) Å from the mean plane defined by the nine constituent atoms. The dihedral angle between the 4-methylphenyl ring and the mean plane of the benzofuran ring is 29.53 (4)°.

In the crystal, molecules are connected by a pair of weak C—H···O hydrogen bonds, forming inversion dimers that stack along the a axis (Table 1).

S2. Experimental

3-Chloroperoxybenzoic acid (77%, 269 mg, 1.2 mmol) was added in small portions to a stirred solution of 5-fluoro-2-(4-methylphenyl)-3-methylsulfanyl-1-benzofuran (326 mg, 1.1 mmol) in dichloromethane (30 mL) at 273 K. After being stirred at room temperature for 4h, 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, 1:1 v/v) to afford the title compound as a colorless solid [yield 73%, m.p. 417–418 K; R_f = 0.45 (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 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 and 0.98 Å for methyl H atoms, respectively. U_{iso}(H) = 1.2U_{eq}(C) for aryl and = 1.5U_{eq}(C) for methyl H atoms. The positions of methyl hydrogens were optimized rotationally.

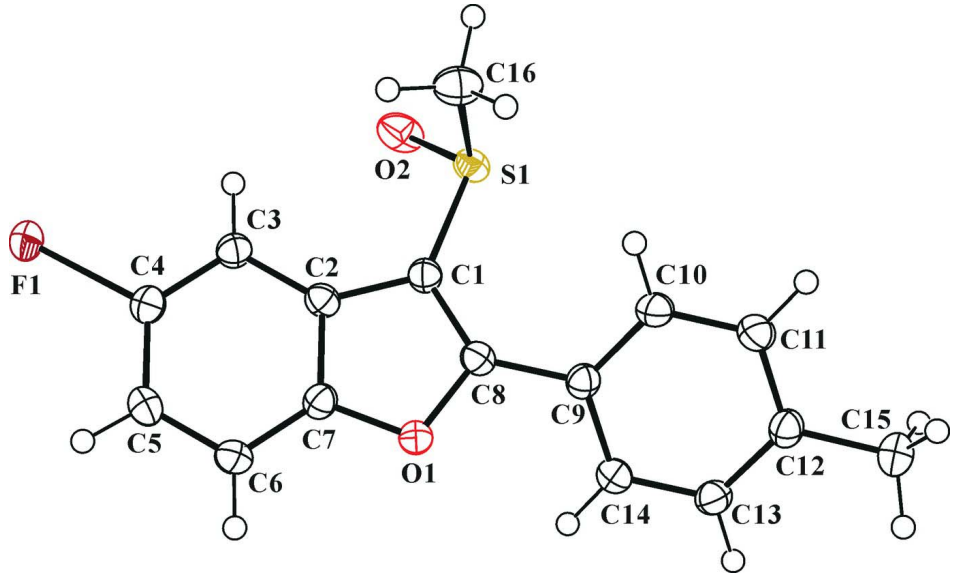

Figure 1

The molecular structure of the title molecule, with the atom numbering. Displacement ellipsoids are drawn at the 50% probability level.

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

Crystal data

$C_{16}H_{13}FO_2S$
$M_r = 288.32$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.0407$ (2) Å
$b = 8.0838$ (2) Å
$c = 11.3517$ (2) Å
$\alpha = 80.126$ (1)°
$\beta = 85.091$ (1)°
$\gamma = 66.773$ (1)°
$V = 667.88$ (3) Å^3
$Z = 2$
$F(000) = 300$
$D_x = 1.434$ Mg m^{-3}
Melting point < 418 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7852 reflections
$\theta = 2.8$–$28.3°$
$\mu = 0.25$ mm^{-1}
$T = 173$ K
Block, colourless
0.53 × 0.40 × 0.24 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)
$T_{min} = 0.701$, $T_{max} = 0.746$
12234 measured reflections
3307 independent reflections
3095 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 28.3°$, $\theta_{min} = 1.8°$
$h = -10 \rightarrow 10$
$k = -10 \rightarrow 10$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.091$
$S = 1.09$
3307 reflections
183 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
$w = 1/[\sigma^2(F_o^2) + (0.0463P)^2 + 0.2225P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.33118 (4)	0.20343 (4)	0.41621 (3)	0.02410 (10)
O1	0.15249 (12)	0.70005 (11)	0.50374 (7)	0.02400 (18)
F1	−0.01539 (13)	0.79204 (12)	0.03932 (7)	0.0428 (2)
O2	0.21524 (13)	0.17958 (13)	0.33018 (10)	0.0378 (2)
C1	0.24601 (16)	0.43759 (15)	0.42709 (10)	0.0221 (2)
C9	0.30709 (16)	0.43979 (16)	0.64794 (10)	0.0223 (2)
C8	0.23981 (16)	0.51353 (15)	0.52688 (10)	0.0221 (2)
C7	0.10062 (16)	0.74208 (16)	0.38628 (10)	0.0226 (2)
C10	0.46136 (17)	0.28077 (17)	0.67089 (11)	0.0260 (2)
H10	0.5246	0.2185	0.6068	0.031*
C2	0.15628 (16)	0.58455 (15)	0.33364 (10)	0.0221 (2)
C14	0.21693 (17)	0.53140 (17)	0.74329 (11)	0.0260 (2)
H14	0.1117	0.6401	0.7292	0.031*
C3	0.11782 (17)	0.59876 (17)	0.21373 (11)	0.0267 (2)
H3	0.1527	0.4950	0.1745	0.032*
C12	0.43513 (19)	0.30326 (18)	0.88157 (11)	0.0294 (3)
C11	0.52340 (18)	0.21267 (18)	0.78664 (11)	0.0292 (3)
H11	0.6274	0.1029	0.8011	0.035*
C6	0.01006 (17)	0.91590 (17)	0.32716 (11)	0.0274 (3)
H6	−0.0244	1.0202	0.3658	0.033*
C13	0.28174 (19)	0.46301 (18)	0.85797 (11)	0.0299 (3)
H13	0.2203	0.5264	0.9221	0.036*
C5	−0.02757 (18)	0.92941 (18)	0.20837 (12)	0.0303 (3)
H5	−0.0899	1.0452	0.1629	0.036*
C16	0.53640 (17)	0.19025 (18)	0.33560 (12)	0.0293 (3)

H16A	0.5951	0.0708	0.3092	0.044*
H16B	0.6178	0.2069	0.3877	0.044*
H16C	0.5085	0.2859	0.2657	0.044*
C4	0.02649 (18)	0.77212 (19)	0.15608 (11)	0.0297 (3)
C15	0.5046 (2)	0.2319 (2)	1.00707 (13)	0.0438 (4)
H15A	0.6136	0.1200	1.0067	0.066*
H15B	0.4114	0.2057	1.0589	0.066*
H15C	0.5341	0.3235	1.0371	0.066*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.02488 (16)	0.01864 (15)	0.02810 (16)	−0.00752 (11)	0.00168 (11)	−0.00507 (11)
O1	0.0276 (4)	0.0198 (4)	0.0228 (4)	−0.0066 (3)	−0.0014 (3)	−0.0045 (3)
F1	0.0568 (6)	0.0438 (5)	0.0221 (4)	−0.0142 (4)	−0.0091 (4)	0.0014 (3)
O2	0.0297 (5)	0.0315 (5)	0.0574 (6)	−0.0110 (4)	−0.0052 (4)	−0.0204 (5)
C1	0.0231 (5)	0.0196 (5)	0.0228 (5)	−0.0074 (4)	0.0002 (4)	−0.0038 (4)
C9	0.0237 (5)	0.0230 (5)	0.0216 (5)	−0.0105 (4)	−0.0004 (4)	−0.0035 (4)
C8	0.0218 (5)	0.0197 (5)	0.0238 (5)	−0.0070 (4)	0.0009 (4)	−0.0037 (4)
C7	0.0227 (5)	0.0229 (5)	0.0221 (5)	−0.0087 (4)	−0.0005 (4)	−0.0037 (4)
C10	0.0249 (6)	0.0278 (6)	0.0240 (6)	−0.0082 (5)	0.0001 (4)	−0.0060 (4)
C2	0.0217 (5)	0.0209 (5)	0.0234 (5)	−0.0081 (4)	0.0006 (4)	−0.0031 (4)
C14	0.0275 (6)	0.0244 (6)	0.0260 (6)	−0.0094 (5)	0.0015 (5)	−0.0060 (4)
C3	0.0301 (6)	0.0275 (6)	0.0231 (5)	−0.0115 (5)	−0.0002 (5)	−0.0047 (4)
C12	0.0354 (7)	0.0332 (6)	0.0232 (6)	−0.0173 (5)	−0.0050 (5)	−0.0020 (5)
C11	0.0267 (6)	0.0289 (6)	0.0290 (6)	−0.0077 (5)	−0.0050 (5)	−0.0017 (5)
C6	0.0271 (6)	0.0214 (5)	0.0310 (6)	−0.0069 (5)	−0.0005 (5)	−0.0033 (5)
C13	0.0362 (7)	0.0327 (6)	0.0233 (6)	−0.0150 (6)	0.0024 (5)	−0.0083 (5)
C5	0.0295 (6)	0.0250 (6)	0.0308 (6)	−0.0070 (5)	−0.0031 (5)	0.0031 (5)
C16	0.0263 (6)	0.0314 (6)	0.0305 (6)	−0.0105 (5)	0.0036 (5)	−0.0091 (5)
C4	0.0324 (6)	0.0342 (7)	0.0210 (5)	−0.0125 (5)	−0.0030 (5)	−0.0002 (5)
C15	0.0576 (10)	0.0467 (9)	0.0260 (7)	−0.0186 (8)	−0.0123 (6)	−0.0012 (6)

Geometric parameters (Å, º)

S1—O2	1.4907 (10)	C3—C4	1.3738 (18)
S1—C1	1.7637 (11)	C3—H3	0.9500
S1—C16	1.7911 (13)	C12—C11	1.3897 (18)
O1—C7	1.3767 (14)	C12—C13	1.3937 (19)
O1—C8	1.3770 (14)	C12—C15	1.5082 (17)
F1—C4	1.3625 (14)	C11—H11	0.9500
C1—C8	1.3665 (16)	C6—C5	1.3839 (18)
C1—C2	1.4422 (16)	C6—H6	0.9500
C9—C10	1.3935 (17)	C13—H13	0.9500
C9—C14	1.4009 (16)	C5—C4	1.3917 (19)
C9—C8	1.4589 (16)	C5—H5	0.9500
C7—C6	1.3814 (16)	C16—H16A	0.9800
C7—C2	1.3945 (16)	C16—H16B	0.9800

C10—C11	1.3878 (17)	C16—H16C	0.9800
C10—H10	0.9500	C15—H15A	0.9800
C2—C3	1.3971 (16)	C15—H15B	0.9800
C14—C13	1.3835 (17)	C15—H15C	0.9800
C14—H14	0.9500		

O2—S1—C1	106.91 (6)	C11—C12—C15	120.89 (13)
O2—S1—C16	105.80 (6)	C13—C12—C15	120.67 (12)
C1—S1—C16	97.50 (6)	C10—C11—C12	120.76 (12)
C7—O1—C8	106.64 (9)	C10—C11—H11	119.6
C8—C1—C2	107.14 (10)	C12—C11—H11	119.6
C8—C1—S1	126.89 (9)	C7—C6—C5	116.19 (11)
C2—C1—S1	125.80 (9)	C7—C6—H6	121.9
C10—C9—C14	118.99 (11)	C5—C6—H6	121.9
C10—C9—C8	121.35 (10)	C14—C13—C12	121.48 (12)
C14—C9—C8	119.65 (11)	C14—C13—H13	119.3
C1—C8—O1	110.59 (10)	C12—C13—H13	119.3
C1—C8—C9	133.89 (11)	C6—C5—C4	119.51 (12)
O1—C8—C9	115.52 (10)	C6—C5—H5	120.2
O1—C7—C6	125.11 (11)	C4—C5—H5	120.2
O1—C7—C2	110.54 (10)	S1—C16—H16A	109.5
C6—C7—C2	124.33 (11)	S1—C16—H16B	109.5
C11—C10—C9	120.55 (11)	H16A—C16—H16B	109.5
C11—C10—H10	119.7	S1—C16—H16C	109.5
C9—C10—H10	119.7	H16A—C16—H16C	109.5
C7—C2—C3	119.30 (11)	H16B—C16—H16C	109.5
C7—C2—C1	105.09 (10)	F1—C4—C3	117.81 (12)
C3—C2—C1	135.61 (11)	F1—C4—C5	117.39 (11)
C13—C14—C9	119.77 (12)	C3—C4—C5	124.80 (12)
C13—C14—H14	120.1	C12—C15—H15A	109.5
C9—C14—H14	120.1	C12—C15—H15B	109.5
C4—C3—C2	115.87 (11)	H15A—C15—H15B	109.5
C4—C3—H3	122.1	C12—C15—H15C	109.5
C2—C3—H3	122.1	H15A—C15—H15C	109.5
C11—C12—C13	118.44 (11)	H15B—C15—H15C	109.5

O2—S1—C1—C8	144.62 (11)	C8—C1—C2—C7	−0.31 (13)
C16—S1—C1—C8	−106.28 (12)	S1—C1—C2—C7	175.22 (9)
O2—S1—C1—C2	−30.03 (12)	C8—C1—C2—C3	179.87 (13)
C16—S1—C1—C2	79.07 (11)	S1—C1—C2—C3	−4.6 (2)
C2—C1—C8—O1	−0.23 (13)	C10—C9—C14—C13	−0.10 (18)
S1—C1—C8—O1	−175.69 (8)	C8—C9—C14—C13	−179.19 (11)
C2—C1—C8—C9	−179.10 (12)	C7—C2—C3—C4	0.33 (17)
S1—C1—C8—C9	5.4 (2)	C1—C2—C3—C4	−179.86 (13)
C7—O1—C8—C1	0.68 (13)	C9—C10—C11—C12	−1.2 (2)
C7—O1—C8—C9	179.78 (9)	C13—C12—C11—C10	0.7 (2)
C10—C9—C8—C1	29.5 (2)	C15—C12—C11—C10	−178.63 (13)
C14—C9—C8—C1	−151.45 (13)	O1—C7—C6—C5	179.08 (11)

C10—C9—C8—O1	−149.35 (11)	C2—C7—C6—C5	0.79 (19)
C14—C9—C8—O1	29.72 (15)	C9—C14—C13—C12	−0.41 (19)
C8—O1—C7—C6	−179.38 (11)	C11—C12—C13—C14	0.1 (2)
C8—O1—C7—C2	−0.89 (13)	C15—C12—C13—C14	179.43 (13)
C14—C9—C10—C11	0.90 (18)	C7—C6—C5—C4	−0.17 (19)
C8—C9—C10—C11	179.98 (11)	C2—C3—C4—F1	−179.35 (11)
O1—C7—C2—C3	−179.40 (10)	C2—C3—C4—C5	0.3 (2)
C6—C7—C2—C3	−0.90 (18)	C6—C5—C4—F1	179.26 (12)
O1—C7—C2—C1	0.74 (13)	C6—C5—C4—C3	−0.4 (2)
C6—C7—C2—C1	179.25 (11)		

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	3.4138 (16)	154

Symmetry code: (i) −*x*, −*y*+1, −*z*+1.