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5-Chloro-3-ethylsulfinyl-2-(4-fluorophenyl)-7-methyl-1-benzofuran

Hong Dae Choi,[a] Pil Ja Seo,[a] Byeng Wha Son[b] and Uk Lee[b]*

[a]Department of Chemistry, Dongeui University, San 24 Kaya-dong Busanjin-gu, Busan 614-714, Republic of Korea, and [b]Department of Chemistry, Pukyong National University, 599-1 Daeyeon 3-dong, Nam-gu, Busan 608-737, Republic of Korea
Correspondence e-mail: uklee@pknu.ac.kr

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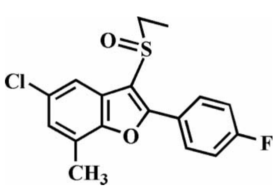
Key indicators: single-crystal X-ray study; T = 174 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.106; data-to-parameter ratio = 17.2.

In the title compound, $C_{17}H_{14}ClFO_2S$, the 4-fluorophenyl ring is rotated slightly out of the benzofuran plane, as indicated by the dihedral angle of 8.32 (5)°. The crystal structure features a short Cl···O contact [3.092 (1) Å].

Related literature

For the crystal structures of similar 3-ethylsulfinyl-2-(4-fluorophenyl)-5-halo-1-benzofuran derivatives, see: Choi *et al.* (2010*a*,*b*,*c*). 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 a review of halogen bonding, see: Politzer *et al.* (2007).



Experimental

Crystal data

$C_{17}H_{14}ClFO_2S$
M_r = 336.79
Triclinic, $P\overline{1}$
a = 7.3395 (1) Å
b = 10.5618 (2) Å
c = 11.2281 (2) Å
α = 65.357 (1)°
β = 85.232 (1)°
γ = 69.939 (1)°
V = 741.28 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.41 mm^{-1}
T = 174 K
0.32 × 0.28 × 0.19 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009) T_{min} = 0.879, T_{max} = 0.926
13016 measured reflections
3440 independent reflections
3292 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.106
S = 1.06
3440 reflections
200 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.59 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: VM2020).

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supporting information

Acta Cryst. (2010). E**66**, o886 [doi:10.1107/S1600536810008627]

5-Chloro-3-ethylsulfinyl-2-(4-fluorophenyl)-7-methyl-1-benzofuran

Hong Dae Choi, Pil Ja Seo, Byeng Wha Son and Uk Lee

S1. Comment

Compounds containing benzofuran moiety show various pharmacological activities such as antifungal (Aslam *et al.*, 2006), antitumor and antiviral (Galal *et al.*, 2009), antimicrobial (Khan *et al.*, 2005) properties. 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 3-ethylsulfinyl-2-(4-fluorophenyl)-5-halo-1-benzofuran analogues (Choi *et al.*, 2010*a,b,c*), we report the crystal structure of the title compound (Fig. 1).

The benzofuran unit is essentially planar, with a mean deviation of 0.010 (1) Å from the least-squares plane defined by the nine constituent atoms. The dihedral angle formed by the benzofuran plane and the 4-fluorophenyl ring is 8.32 (5)°. The crystal packing (Fig. 2) is stabilized by a Cl···O halogen bond between the chlorine and the oxygen of the S═O unit [Cl···O2^{i} = 3.092 (1) Å; C4–Cl···O2^{i} = 167.20 (6)°, symmetry code (i) - *x*, - *y* + 1, - *z*] (Politzer *et al.*, 2007).

S2. Experimental

77% 3-Chloroperoxybenzoic acid (202 mg, 0.9 mmol) was added in small portions to a stirred solution of 5-chloro-3-ethylsulfanyl-2-(4-fluorophenyl)-7-methyl-1-benzofuran (256 mg, 0.8 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 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 83%, m.p. 409-410 K; R_f = 0.64 (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, 0.99 Å for methylene, and 0.98 Å for methyl H atoms. U_{iso}(H) = 1.2U_{eq}(C) for aryl and methylene H atoms, and 1.5U_{eq}(C) for methyl H atoms.

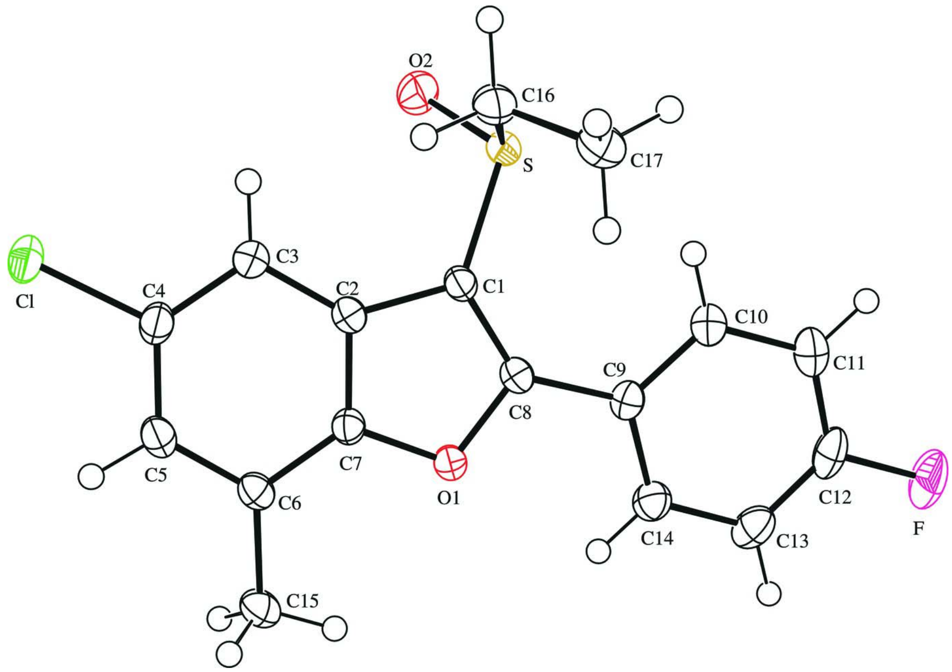

Figure 1

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.

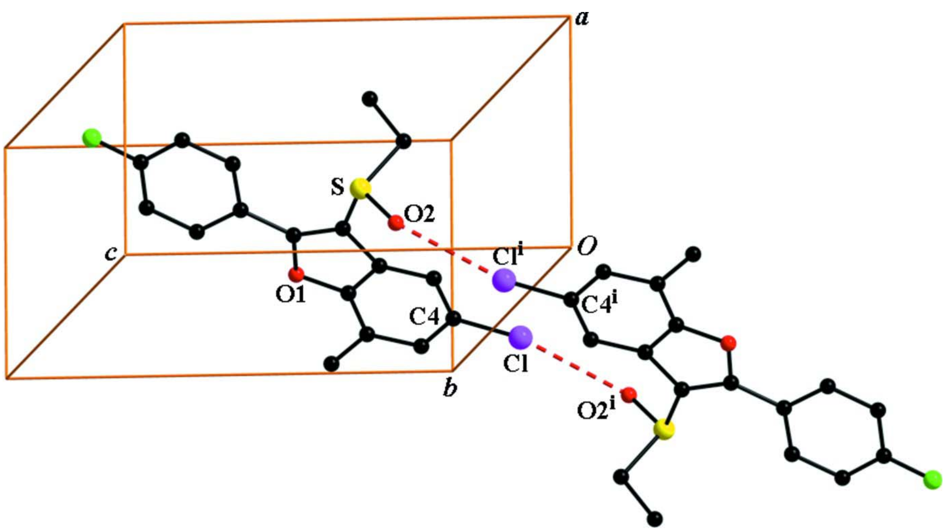

Figure 2

C–Cl···O interactions (dotted lines) in the crystal structure of the title compound. [Symmetry codes: i) - *x*, - *y* + 1, - *z*.]

5-Chloro-3-ethylsulfinyl-2-(4-fluorophenyl)-7-methyl-1-benzofuran

Crystal data

$C_{17}H_{14}ClFO_2S$	a = 7.3395 (1) Å
M_r = 336.79	b = 10.5618 (2) Å
Triclinic, $P\overline{1}$	c = 11.2281 (2) Å
Hall symbol: -P 1	α = 65.357 (1)°

β = 85.232 (1)°
γ = 69.939 (1)°
V = 741.28 (2) Å^3
Z = 2
F(000) = 348
D_x = 1.509 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9795 reflections
θ = 2.3–27.6°
μ = 0.41 mm^{-1}
T = 174 K
Block, colourless
0.32 × 0.28 × 0.19 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Radiation source: Rotating Anode
Bruker HELIOS graded multilayer optics monochromator
Detector resolution: 10.0 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.879, T_{max} = 0.926
13016 measured reflections
3440 independent reflections
3292 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 27.6°, θ_{min} = 2.0°
$h = -9 \rightarrow 9$
$k = -13 \rightarrow 13$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.106
S = 1.06
3440 reflections
200 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
$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2 + 0.4567P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.59 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}
Cl	−0.00786 (7)	0.72104 (5)	−0.07738 (4)	0.03543 (13)
S	0.36819 (5)	0.18932 (4)	0.42209 (3)	0.02170 (12)
F	0.58849 (19)	0.15390 (14)	1.03317 (10)	0.0457 (3)
O1	0.20083 (15)	0.56779 (11)	0.46554 (10)	0.0215 (2)
O2	0.21568 (18)	0.18590 (14)	0.34446 (12)	0.0329 (3)
C1	0.2932 (2)	0.37130 (16)	0.41330 (14)	0.0197 (3)
C2	0.1983 (2)	0.50252 (16)	0.29663 (14)	0.0202 (3)
C3	0.1543 (2)	0.53059 (17)	0.16738 (15)	0.0230 (3)
H3	0.1893	0.4537	0.1379	0.028*

C4	0.0568 (2)	0.67697 (18)	0.08464 (15)	0.0245 (3)
C5	0.0001 (2)	0.79229 (17)	0.12610 (15)	0.0242 (3)
H5	−0.0683	0.8904	0.0652	0.029*
C6	0.0420 (2)	0.76611 (16)	0.25427 (15)	0.0220 (3)
C7	0.1436 (2)	0.61881 (16)	0.33529 (14)	0.0200 (3)
C8	0.2915 (2)	0.41644 (16)	0.51177 (14)	0.0199 (3)
C9	0.3662 (2)	0.34377 (17)	0.64954 (14)	0.0213 (3)
C10	0.4879 (2)	0.19527 (18)	0.70685 (15)	0.0259 (3)
H10	0.5212	0.1385	0.6563	0.031*
C11	0.5603 (2)	0.13041 (19)	0.83696 (16)	0.0301 (3)
H11	0.6407	0.0292	0.8765	0.036*
C12	0.5129 (3)	0.2158 (2)	0.90716 (15)	0.0315 (4)
C13	0.3926 (3)	0.3615 (2)	0.85540 (16)	0.0318 (4)
H13	0.3610	0.4171	0.9071	0.038*
C14	0.3184 (2)	0.42539 (18)	0.72601 (16)	0.0266 (3)
H14	0.2341	0.5256	0.6888	0.032*
C15	−0.0198 (2)	0.88632 (18)	0.30237 (17)	0.0281 (3)
H15A	−0.1619	0.9314	0.2920	0.034*
H15B	0.0382	0.9620	0.2512	0.034*
H15C	0.0238	0.8442	0.3953	0.034*
C16	0.5748 (2)	0.19716 (18)	0.32281 (16)	0.0278 (3)
H16A	0.6081	0.1195	0.2891	0.033*
H16B	0.5414	0.2947	0.2466	0.033*
C17	0.7481 (2)	0.17387 (19)	0.40416 (17)	0.0296 (3)
H17A	0.7134	0.2489	0.4397	0.044*
H17B	0.8574	0.1827	0.3487	0.044*
H17C	0.7855	0.0750	0.4767	0.044*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0530 (3)	0.0313 (2)	0.0190 (2)	−0.01523 (19)	−0.00769 (17)	−0.00508 (16)
S	0.0252 (2)	0.01716 (18)	0.02138 (19)	−0.00641 (14)	−0.00133 (14)	−0.00688 (14)
F	0.0555 (7)	0.0562 (7)	0.0189 (5)	−0.0214 (6)	−0.0094 (5)	−0.0055 (5)
O1	0.0236 (5)	0.0192 (5)	0.0188 (5)	−0.0035 (4)	−0.0010 (4)	−0.0077 (4)
O2	0.0357 (6)	0.0309 (6)	0.0355 (7)	−0.0144 (5)	−0.0072 (5)	−0.0127 (5)
C1	0.0193 (6)	0.0173 (6)	0.0194 (6)	−0.0039 (5)	−0.0003 (5)	−0.0063 (5)
C2	0.0186 (6)	0.0187 (7)	0.0209 (7)	−0.0049 (5)	0.0005 (5)	−0.0070 (5)
C3	0.0264 (7)	0.0218 (7)	0.0205 (7)	−0.0076 (6)	−0.0003 (5)	−0.0085 (6)
C4	0.0278 (7)	0.0259 (8)	0.0185 (7)	−0.0104 (6)	−0.0024 (6)	−0.0063 (6)
C5	0.0234 (7)	0.0197 (7)	0.0241 (7)	−0.0062 (6)	−0.0029 (6)	−0.0040 (6)
C6	0.0192 (6)	0.0192 (7)	0.0245 (7)	−0.0044 (5)	0.0002 (5)	−0.0078 (6)
C7	0.0189 (6)	0.0204 (7)	0.0187 (6)	−0.0049 (5)	−0.0002 (5)	−0.0074 (5)
C8	0.0182 (6)	0.0183 (6)	0.0206 (7)	−0.0047 (5)	0.0011 (5)	−0.0067 (5)
C9	0.0206 (6)	0.0242 (7)	0.0186 (7)	−0.0098 (6)	0.0013 (5)	−0.0068 (6)
C10	0.0242 (7)	0.0267 (8)	0.0236 (7)	−0.0069 (6)	−0.0013 (6)	−0.0083 (6)
C11	0.0269 (8)	0.0306 (8)	0.0242 (8)	−0.0090 (6)	−0.0033 (6)	−0.0033 (6)
C12	0.0336 (8)	0.0425 (10)	0.0165 (7)	−0.0195 (7)	−0.0027 (6)	−0.0043 (7)

C13	0.0400 (9)	0.0387 (9)	0.0233 (8)	−0.0198 (8)	0.0040 (7)	−0.0143 (7)
C14	0.0315 (8)	0.0269 (8)	0.0218 (7)	−0.0120 (6)	0.0024 (6)	−0.0089 (6)
C15	0.0292 (8)	0.0204 (7)	0.0297 (8)	−0.0016 (6)	0.0003 (6)	−0.0109 (6)
C16	0.0284 (8)	0.0271 (8)	0.0257 (8)	−0.0038 (6)	0.0041 (6)	−0.0137 (6)
C17	0.0260 (7)	0.0267 (8)	0.0358 (9)	−0.0082 (6)	0.0036 (6)	−0.0136 (7)

Geometric parameters (Å, º)

Cl—O2^{i}	3.092 (1)	C9—C14	1.401 (2)
Cl—C4	1.743 (2)	C9—C10	1.402 (2)
S—O2	1.493 (1)	C10—C11	1.389 (2)
S—C1	1.770 (2)	C10—H10	0.9500
S—C16	1.810 (2)	C11—C12	1.374 (3)
F—C12	1.359 (2)	C11—H11	0.9500
O1—C7	1.377 (2)	C12—C13	1.376 (3)
O1—C8	1.380 (2)	C13—C14	1.387 (2)
C1—C8	1.372 (2)	C13—H13	0.9500
C1—C2	1.448 (2)	C14—H14	0.9500
C2—C7	1.391 (2)	C15—H15A	0.9800
C2—C3	1.397 (2)	C15—H15B	0.9800
C3—C4	1.385 (2)	C15—H15C	0.9800
C3—H3	0.9500	C16—C17	1.518 (2)
C4—C5	1.402 (2)	C16—H16A	0.9900
C5—C6	1.388 (2)	C16—H16B	0.9900
C5—H5	0.9500	C17—H17A	0.9800
C6—C7	1.393 (2)	C17—H17B	0.9800
C6—C15	1.498 (2)	C17—H17C	0.9800
C8—C9	1.465 (2)		
C4—Cl—O2^{i}	167.20 (6)	C11—C10—H10	119.7
O2—S—C1	106.41 (7)	C9—C10—H10	119.7
O2—S—C16	107.18 (8)	C12—C11—C10	118.53 (16)
C1—S—C16	97.87 (7)	C12—C11—H11	120.7
C7—O1—C8	106.97 (11)	C10—C11—H11	120.7
C8—C1—C2	107.05 (12)	F—C12—C11	118.63 (16)
C8—C1—S	129.11 (11)	F—C12—C13	118.49 (16)
C2—C1—S	123.49 (11)	C11—C12—C13	122.88 (15)
C7—C2—C3	119.80 (13)	C12—C13—C14	118.44 (16)
C7—C2—C1	105.17 (13)	C12—C13—H13	120.8
C3—C2—C1	135.02 (14)	C14—C13—H13	120.8
C4—C3—C2	116.20 (14)	C13—C14—C9	120.75 (15)
C4—C3—H3	121.9	C13—C14—H14	119.6
C2—C3—H3	121.9	C9—C14—H14	119.6
C3—C4—C5	123.12 (14)	C6—C15—H15A	109.5
C3—C4—Cl	119.13 (12)	C6—C15—H15B	109.5
C5—C4—Cl	117.71 (12)	H15A—C15—H15B	109.5
C6—C5—C4	121.36 (14)	C6—C15—H15C	109.5
C6—C5—H5	119.3	H15A—C15—H15C	109.5

C4—C5—H5	119.3	H15B—C15—H15C	109.5
C5—C6—C7	114.70 (13)	C17—C16—S	110.04 (11)
C5—C6—C15	122.68 (14)	C17—C16—H16A	109.7
C7—C6—C15	122.61 (14)	S—C16—H16A	109.7
O1—C7—C2	110.59 (12)	C17—C16—H16B	109.7
O1—C7—C6	124.61 (13)	S—C16—H16B	109.7
C2—C7—C6	124.79 (14)	H16A—C16—H16B	108.2
C1—C8—O1	110.21 (12)	C16—C17—H17A	109.5
C1—C8—C9	135.48 (13)	C16—C17—H17B	109.5
O1—C8—C9	114.29 (12)	H17A—C17—H17B	109.5
C14—C9—C10	118.79 (14)	C16—C17—H17C	109.5
C14—C9—C8	119.28 (14)	H17A—C17—H17C	109.5
C10—C9—C8	121.92 (13)	H17B—C17—H17C	109.5
C11—C10—C9	120.58 (15)		

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