

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2-(4-Chlorophenyl)-5-fluoro-3-methylsulfinyl-1-benzofuran

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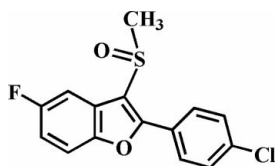
Received 4 January 2010; accepted 10 January 2010

Key indicators: single-crystal X-ray study; *T* = 100 K; mean $\sigma(\text{C}-\text{C})$ = 0.002 Å; *R* factor = 0.032; *wR* factor = 0.088; data-to-parameter ratio = 16.6.

In the title compound, C₁₅H₁₀ClFO₂S, the O atom and the methyl group of the methylsulfinyl substituent are located on opposite sides of the plane through the benzofuran fragment. The 4-chlorophenyl ring is rotated out of the benzofuran plane, as indicated by the dihedral angle of 21.04 (4)°. The crystal structure exhibits a weak intermolecular C—H...O hydrogen bond and a Cl...O halogen bond [Cl...O = 3.254 (1) Å].

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,c*). For the biological properties of benzofuran compounds, see: Aslam *et al.* (2006); Galal *et al.* (2009); Howlett *et al.* (1999). 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

C₁₅H₁₀ClFO₂S
M_r = 308.74

Triclinic, *P* $\bar{1}$
a = 7.7088 (5) Å

b = 8.5241 (5) Å
c = 11.6369 (7) Å
 α = 74.572 (3)°
 β = 70.893 (3)°
 γ = 67.109 (3)°
V = 657.13 (7) Å³

Z = 2
Mo *K*α radiation
 μ = 0.46 mm⁻¹
T = 100 K
0.25 × 0.22 × 0.13 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
*T*_{min} = 0.895, *T*_{max} = 0.942

10607 measured reflections
3020 independent reflections
2629 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.032

Refinement

R[*F*² > 2σ(*F*²)] = 0.032
wR(*F*²) = 0.088
S = 1.03
3020 reflections

182 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}}$ = 0.42 e Å⁻³
 $\Delta\rho_{\text{min}}$ = -0.27 e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C10—H10...O2 ⁱ	0.93	2.52	3.347 (2)	148

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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: VM2017).

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supplementary materials

Acta Cryst. (2010). E66, o364 [doi:10.1107/S1600536810001157]

2-(4-Chlorophenyl)-5-fluoro-3-methylsulfinyl-1-benzofuran

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Comment

Benzofuran ring systems have drawn considerable interest in view of their biological properties (Aslam *et al.*, 2006; Galal *et al.*, 2009; Howlett *et al.* 1999) and their occurrence as natural products (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,c*), we report the crystal structure of the title compound (Fig. 1).

The benzofuran unit is essentially planar, with a mean deviation of 0.006 (1) Å from the least-squares plane defined by the nine constituent atoms. The dihedral angle formed by the plane of the benzofuran and the 4-chlorophenyl ring is 21.04 (4)°. The crystal packing (Fig. 2) is stabilized by a weak intermolecular C–H···O hydrogen bond between the 4-chlorophenyl H atom and the oxygen of the S=O unit (Table 1). The molecular packing (Fig. 2) is further stabilized by a Cl···O halogen bond between the chlorine and the oxygen of the S=O unit [$\text{Cl}\cdots\text{O}2^{\text{ii}} = 3.254 (1) \text{ \AA}$; $\text{C}-\text{Cl}\cdots\text{O}2 = 163.42 (6)^\circ$] (Politzer *et al.*, 2007).

Experimental

77% 3-Chloroperoxybenzoic acid (291 mg, 1.3 mmol) was added in small portions to a stirred solution of 2-(4-chlorophenyl)-5-fluoro-3-methylsulfonyl-1-benzofuran (351 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, 2:1 v/v) to afford the title compound as a colorless solid [yield 81 %, m.p. 442–443 K; $R_f = 0.41$ (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 benzene at room temperature.

Refinement

All H atoms were positioned geometrically and refined using a riding model, with C–H = 0.93 Å for aromatic H atoms and 0.96 Å for methyl H atoms, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic H atoms and $1.55U_{\text{eq}}(\text{C})$ for methyl H atoms.

Figures

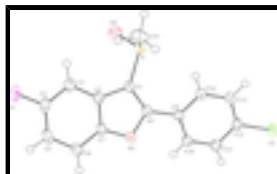


Fig. 1. The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as a small spheres of arbitrary radius.

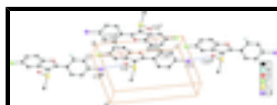


Fig. 2. C–H···O and C–Cl···O interactions (dotted lines) in the crystal structure of the title compound. [Symmetry codes: (i) $-x, -y + 1, -z + 1$; (ii) $x, y, z + 1$; (iii) $x, y, z - 1$.]

2-(4-Chlorophenyl)-5-fluoro-3-methylsulfinyl-1-benzofuran

Crystal data

$C_{15}H_{10}ClFO_2S$	$Z = 2$
$M_r = 308.74$	$F(000) = 316$
Triclinic, PT	$D_x = 1.560 \text{ Mg m}^{-3}$
Hall symbol: $-P 1$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 7.7088 (5) \text{ \AA}$	Cell parameters from 6597 reflections
$b = 8.5241 (5) \text{ \AA}$	$\theta = 2.6\text{--}27.6^\circ$
$c = 11.6369 (7) \text{ \AA}$	$\mu = 0.46 \text{ mm}^{-1}$
$\alpha = 74.572 (3)^\circ$	$T = 100 \text{ K}$
$\beta = 70.893 (3)^\circ$	Block, colourless
$\gamma = 67.109 (3)^\circ$	$0.25 \times 0.22 \times 0.13 \text{ mm}$
$V = 657.13 (7) \text{ \AA}^3$	

Data collection

Bruker SMART APEXII CCD diffractometer	3020 independent reflections
Radiation source: Rotating Anode HELIOS	2629 reflections with $I > 2\sigma(I)$
Detector resolution: $10.0 \text{ pixels mm}^{-1}$	$R_{\text{int}} = 0.032$
φ and ω scans	$\theta_{\text{max}} = 27.6^\circ$, $\theta_{\text{min}} = 1.9^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2009)	$h = -9 \rightarrow 10$
$T_{\text{min}} = 0.895$, $T_{\text{max}} = 0.942$	$k = -11 \rightarrow 11$
10607 measured reflections	$l = -15 \rightarrow 15$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.032$	Hydrogen site location: difference Fourier map
$wR(F^2) = 0.088$	H-atom parameters constrained
$S = 1.02$	$w = 1/[\sigma^2(F_o^2) + (0.0428P)^2 + 0.2892P]$
3020 reflections	where $P = (F_o^2 + 2F_c^2)/3$
182 parameters	$(\Delta/\sigma)_{\text{max}} < 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 0.42 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.27 \text{ e \AA}^{-3}$

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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl	0.33113 (7)	0.22955 (5)	1.02698 (4)	0.03627 (12)
S	0.35910 (5)	0.20350 (5)	0.40485 (3)	0.02555 (11)
F	0.16308 (18)	0.76845 (15)	0.01238 (10)	0.0474 (3)
O1	0.17250 (15)	0.67346 (13)	0.48816 (10)	0.0258 (2)
O2	0.25799 (18)	0.18174 (15)	0.32408 (12)	0.0356 (3)
C1	0.2811 (2)	0.42581 (18)	0.41217 (13)	0.0232 (3)
C2	0.2278 (2)	0.56740 (19)	0.31515 (14)	0.0243 (3)
C3	0.2310 (2)	0.5825 (2)	0.19235 (14)	0.0299 (3)
H3	0.2760	0.4867	0.1534	0.036*
C4	0.1632 (3)	0.7478 (2)	0.13242 (15)	0.0325 (4)
C5	0.0958 (3)	0.8951 (2)	0.18489 (16)	0.0339 (4)
H5	0.0520	1.0032	0.1387	0.041*
C6	0.0940 (2)	0.8808 (2)	0.30654 (15)	0.0302 (3)
H6	0.0506	0.9771	0.3446	0.036*
C7	0.1604 (2)	0.7151 (2)	0.36803 (13)	0.0248 (3)
C8	0.2452 (2)	0.49619 (18)	0.51340 (13)	0.0231 (3)
C9	0.2682 (2)	0.42787 (18)	0.63814 (13)	0.0225 (3)
C10	0.1603 (2)	0.52936 (19)	0.73078 (14)	0.0251 (3)
H10	0.0727	0.6383	0.7124	0.030*
C12	0.3088 (2)	0.3053 (2)	0.87660 (13)	0.0258 (3)
C11	0.1826 (2)	0.4693 (2)	0.84935 (14)	0.0277 (3)
H11	0.1134	0.5386	0.9102	0.033*
C13	0.4161 (2)	0.2011 (2)	0.78744 (14)	0.0277 (3)
H13	0.4990	0.0903	0.8073	0.033*
C14	0.3979 (2)	0.2645 (2)	0.66813 (14)	0.0269 (3)
H14	0.4734	0.1971	0.6067	0.032*
C15	0.6025 (2)	0.1816 (2)	0.31476 (18)	0.0396 (4)
H15A	0.6681	0.0656	0.2979	0.059*
H15B	0.6705	0.2072	0.3594	0.059*
H15C	0.5994	0.2603	0.2385	0.059*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0509 (3)	0.0319 (2)	0.0236 (2)	-0.00862 (18)	-0.01631 (17)	0.00016 (15)
S	0.0298 (2)	0.01997 (19)	0.0270 (2)	-0.00747 (15)	-0.00886 (15)	-0.00284 (14)
F	0.0699 (8)	0.0475 (7)	0.0278 (5)	-0.0194 (6)	-0.0255 (5)	0.0055 (4)

supplementary materials

O1	0.0325 (6)	0.0201 (5)	0.0237 (5)	-0.0059 (4)	-0.0107 (4)	-0.0013 (4)
O2	0.0393 (6)	0.0307 (6)	0.0433 (7)	-0.0091 (5)	-0.0178 (5)	-0.0107 (5)
C1	0.0264 (7)	0.0195 (7)	0.0237 (7)	-0.0073 (6)	-0.0084 (5)	-0.0009 (5)
C2	0.0262 (7)	0.0227 (7)	0.0254 (7)	-0.0095 (6)	-0.0095 (6)	0.0003 (6)
C3	0.0373 (8)	0.0306 (8)	0.0252 (7)	-0.0135 (7)	-0.0111 (6)	-0.0025 (6)
C4	0.0391 (9)	0.0381 (9)	0.0227 (7)	-0.0154 (7)	-0.0151 (6)	0.0046 (6)
C5	0.0374 (9)	0.0282 (8)	0.0347 (9)	-0.0110 (7)	-0.0181 (7)	0.0087 (6)
C6	0.0335 (8)	0.0219 (7)	0.0340 (8)	-0.0065 (6)	-0.0135 (6)	-0.0006 (6)
C7	0.0267 (7)	0.0255 (7)	0.0234 (7)	-0.0093 (6)	-0.0100 (6)	-0.0003 (6)
C8	0.0230 (7)	0.0195 (7)	0.0256 (7)	-0.0058 (5)	-0.0080 (5)	-0.0013 (5)
C9	0.0245 (7)	0.0223 (7)	0.0224 (7)	-0.0092 (6)	-0.0081 (5)	-0.0013 (5)
C10	0.0261 (7)	0.0216 (7)	0.0258 (7)	-0.0060 (6)	-0.0079 (6)	-0.0019 (5)
C12	0.0301 (7)	0.0279 (8)	0.0211 (7)	-0.0114 (6)	-0.0099 (6)	0.0005 (6)
C11	0.0304 (8)	0.0274 (8)	0.0240 (7)	-0.0091 (6)	-0.0046 (6)	-0.0055 (6)
C13	0.0307 (8)	0.0229 (7)	0.0276 (8)	-0.0043 (6)	-0.0130 (6)	-0.0010 (6)
C14	0.0295 (8)	0.0248 (7)	0.0253 (7)	-0.0049 (6)	-0.0090 (6)	-0.0058 (6)
C15	0.0306 (8)	0.0324 (9)	0.0527 (11)	-0.0088 (7)	-0.0035 (8)	-0.0131 (8)

Geometric parameters (Å, °)

Cl—O2 ⁱ	3.254 (1)	C6—C7	1.382 (2)
Cl—C12	1.737 (2)	C6—H6	0.9300
S—O2	1.487 (1)	C8—C9	1.455 (2)
S—C1	1.768 (2)	C9—C14	1.396 (2)
S—C15	1.788 (2)	C9—C10	1.397 (2)
F—C4	1.361 (2)	C10—C11	1.381 (2)
O1—C7	1.373 (2)	C10—H10	0.9300
O1—C8	1.377 (2)	C12—C13	1.381 (2)
C1—C8	1.363 (2)	C12—C11	1.382 (2)
C1—C2	1.447 (2)	C11—H11	0.9300
C2—C7	1.388 (2)	C13—C14	1.381 (2)
C2—C3	1.393 (2)	C13—H13	0.9300
C3—C4	1.374 (2)	C14—H14	0.9300
C3—H3	0.9300	C15—H15A	0.9600
C4—C5	1.382 (3)	C15—H15B	0.9600
C5—C6	1.384 (2)	C15—H15C	0.9600
C5—H5	0.9300		
C12—Cl—O2 ⁱ	163.42 (6)	C1—C8—C9	134.98 (14)
O2—S—C1	107.93 (7)	O1—C8—C9	114.44 (12)
O2—S—C15	105.99 (8)	C14—C9—C10	118.63 (13)
C1—S—C15	97.60 (8)	C14—C9—C8	122.01 (13)
C7—O1—C8	106.67 (11)	C10—C9—C8	119.35 (13)
C8—C1—C2	106.99 (13)	C11—C10—C9	120.57 (14)
C8—C1—S	126.32 (11)	C11—C10—H10	119.7
C2—C1—S	126.50 (11)	C9—C10—H10	119.7
C7—C2—C3	119.55 (14)	C13—C12—C11	121.54 (14)
C7—C2—C1	105.05 (13)	C13—C12—Cl	119.65 (12)
C3—C2—C1	135.40 (14)	C11—C12—Cl	118.80 (12)

C4—C3—C2	115.73 (15)	C10—C11—C12	119.31 (14)
C4—C3—H3	122.1	C10—C11—H11	120.3
C2—C3—H3	122.1	C12—C11—H11	120.3
F—C4—C3	117.68 (15)	C12—C13—C14	118.74 (14)
F—C4—C5	117.49 (15)	C12—C13—H13	120.6
C3—C4—C5	124.84 (15)	C14—C13—H13	120.6
C4—C5—C6	119.63 (15)	C13—C14—C9	121.16 (14)
C4—C5—H5	120.2	C13—C14—H14	119.4
C6—C5—H5	120.2	C9—C14—H14	119.4
C7—C6—C5	116.06 (15)	S—C15—H15A	109.5
C7—C6—H6	122.0	S—C15—H15B	109.5
C5—C6—H6	122.0	H15A—C15—H15B	109.5
O1—C7—C6	125.09 (14)	S—C15—H15C	109.5
O1—C7—C2	110.71 (13)	H15A—C15—H15C	109.5
C6—C7—C2	124.19 (14)	H15B—C15—H15C	109.5
C1—C8—O1	110.57 (13)		
O2—S—C1—C8	141.66 (14)	C1—C2—C7—C6	180.00 (14)
C15—S—C1—C8	-108.75 (15)	C2—C1—C8—O1	-0.06 (17)
O2—S—C1—C2	-32.73 (15)	S—C1—C8—O1	-175.34 (10)
C15—S—C1—C2	76.86 (15)	C2—C1—C8—C9	-179.09 (16)
C8—C1—C2—C7	-0.66 (16)	S—C1—C8—C9	5.6 (3)
S—C1—C2—C7	174.61 (11)	C7—O1—C8—C1	0.77 (16)
C8—C1—C2—C3	179.28 (17)	C7—O1—C8—C9	-179.99 (12)
S—C1—C2—C3	-5.5 (3)	C1—C8—C9—C14	22.0 (3)
C7—C2—C3—C4	-0.5 (2)	O1—C8—C9—C14	-156.97 (14)
C1—C2—C3—C4	179.56 (16)	C1—C8—C9—C10	-159.18 (17)
C2—C3—C4—F	-179.79 (14)	O1—C8—C9—C10	21.82 (19)
C2—C3—C4—C5	0.5 (3)	C14—C9—C10—C11	0.3 (2)
F—C4—C5—C6	-179.70 (15)	C8—C9—C10—C11	-178.49 (13)
C3—C4—C5—C6	0.0 (3)	C9—C10—C11—C12	-1.9 (2)
C4—C5—C6—C7	-0.5 (2)	C13—C12—C11—C10	1.3 (2)
C8—O1—C7—C6	179.96 (14)	Cl—C12—C11—C10	-178.53 (12)
C8—O1—C7—C2	-1.21 (16)	C11—C12—C13—C14	0.9 (2)
C5—C6—C7—O1	179.14 (14)	Cl—C12—C13—C14	-179.24 (12)
C5—C6—C7—C2	0.5 (2)	C12—C13—C14—C9	-2.6 (2)
C3—C2—C7—O1	-178.79 (13)	C10—C9—C14—C13	1.9 (2)
C1—C2—C7—O1	1.16 (16)	C8—C9—C14—C13	-179.25 (14)
C3—C2—C7—C6	0.0 (2)		

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C10—H10 ⁱⁱ ⋯O2 ⁱⁱ	0.93	2.52	3.347 (2)	148

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

Fig. 1

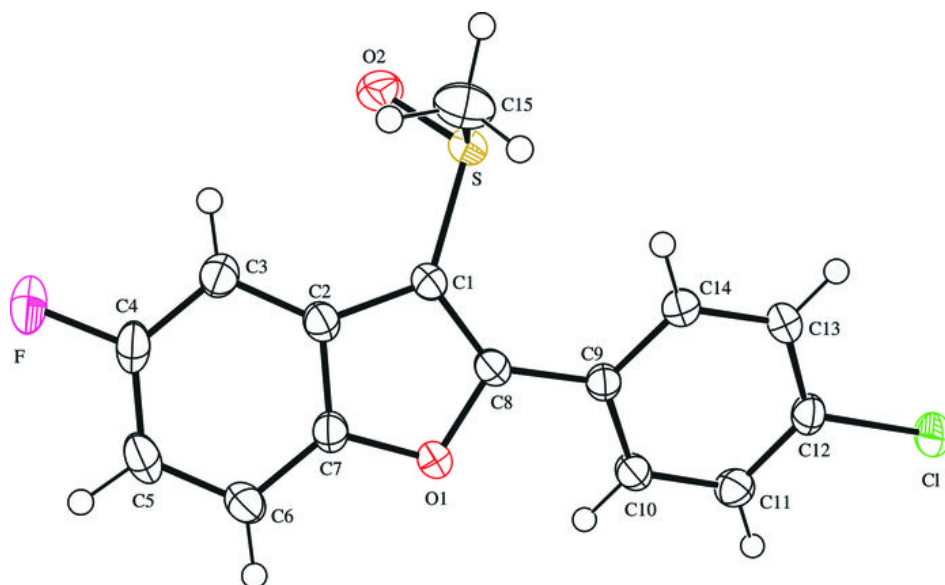


Fig. 2

