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

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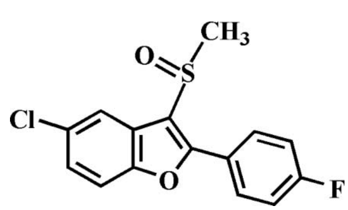
Key indicators: single-crystal X-ray study; T = 151 K; mean σ(C–C) = 0.002 Å; R factor = 0.029; wR factor = 0.080; data-to-parameter ratio = 16.2.

In the title compound, $C_{15}H_{10}ClFO_2S$, 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-fluorophenyl ring is rotated out of the benzofuran plane, making a dihedral angle of 25.99 (4)°. The crystal structure is stabilized by a non-classical intermolecular C—H···O hydrogen bond and a Cl···O halogen bond [3.244 (1) Å].

Related literature

For the crystal structures of similar 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). For a review of halogen bonding, see: Politzer *et al.* (2007).



Experimental

Crystal data

$C_{15}H_{10}ClFO_2S$
M_r = 308.74
Triclinic, $P\overline{1}$
a = 7.8484 (2) Å
b = 8.3176 (2) Å
c = 10.7353 (2) Å
α = 95.637 (1)°
β = 91.975 (1)°
γ = 112.372 (1)°
V = 642.95 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.47 mm^{-1}
T = 151 K
0.38 × 0.37 × 0.31 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.843, T_{max} = 0.868
11361 measured reflections
2949 independent reflections
2799 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.080
S = 1.06
2949 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 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	2.56	3.4287 (16)	152

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

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: ZQ2011).

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supplementary materials

Acta Cryst. (2009). E**65**, o2649 [doi:10.1107/S1600536809039749]

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

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Comment

Molecules involving benzofuran moiety have received considerable attention owing to a variety of their 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 continuing studies of the effect of side chain substituents on the solid state structures of 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 0.008 (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 25.99 (4)°. The crystal packing (Fig. 2) is stabilized by a non-classical intermolecular C—H···O hydrogen bond between the 4-fluorophenyl H atom and the oxygen of the S═O unit, with a C14—H14···O2^{i} (Table 1), and a Cl···O halogen bond between the chlorine and the oxygen of the S═O unit [Cl···O2^{ii} = 3.244 (1) Å; C—Cl···O = 174.59 (5) °] (Politzer *et al.*, 2007).

Experimental

77% 3-Chloroperoxybenzoic acid (291 mg, 1.3 mmol) was added in small portions to a stirred solution of 5-chloro-2-(4-fluorophenyl)-3-methylsulfanyl-1-benzofuran (370 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 86%, m.p. 492–493 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 chloroforrm at room temperature.

Refinement

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

Figures

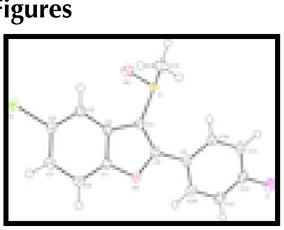


Fig. 1. The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as a small spheres of arbitrary radius.

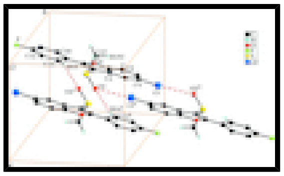


Fig. 2. C—H···O and C—Cl···O interactions (dotted lines) in the crystal structure of the title compound. [Symmetry codes: (i) - x + 1, - y + 1, - z + 1; (ii) - x + 1, - y + 1, - z + 2.]

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

Crystal data

$C_{15}H_{10}ClFO_2S$	$Z = 2$
$M_r = 308.74$	$F_{000} = 316$
Triclinic, $P\overline{1}$	$D_x = 1.595$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 7.8484$ (2) Å	Cell parameters from 8966 reflections
$b = 8.3176$ (2) Å	$\theta = 2.7$–27.6º
$c = 10.7353$ (2) Å	$\mu = 0.47$ mm^{-1}
$\alpha = 95.637$ (1)º	$T = 151$ K
$\beta = 91.975$ (1)º	Block, colourless
$\gamma = 112.372$ (1)º	0.38 × 0.37 × 0.31 mm
$V = 642.95$ (3) Å^3	

Data collection

Bruker SMART APEXII CCD diffractometer	2949 independent reflections
Radiation source: Rotating Anode	2799 reflections with $I > 2\sigma(I)$
Monochromator: HELIOS	$R_{int} = 0.023$
Detector resolution: 10.0 pixels mm^{-1}	$\theta_{max} = 27.6$º
$T = 151$ K	$\theta_{min} = 1.9$º
φ and ω scans	$h = -10 \rightarrow 10$
Absorption correction: multi-scan (SADABS; Bruker, 2009)	$k = -10 \rightarrow 10$
$T_{min} = 0.843$, $T_{max} = 0.868$	$l = -12 \rightarrow 13$
11361 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.029$	H-atom parameters constrained
$wR(F^2) = 0.080$	$w = 1/[\sigma^2(F_o^2) + (0.043P)^2 + 0.2884P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
2949 reflections	$\Delta\rho_{max} = 0.33$ e Å^{-3}
182 parameters	$\Delta\rho_{min} = -0.36$ e Å^{-3}

Primary atom site location: structure-invariant direct methods	Extinction correction: none

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	0.18454 (4)	0.20468 (4)	0.59366 (3)	0.01959 (10)
Cl	0.58536 (5)	0.78256 (4)	1.01739 (3)	0.02696 (10)
F	−0.06796 (13)	0.22320 (12)	−0.01528 (8)	0.0373 (2)
O1	0.33377 (12)	0.68122 (11)	0.48927 (8)	0.01984 (19)
O2	0.32352 (14)	0.18415 (13)	0.68194 (10)	0.0292 (2)
C1	0.25897 (17)	0.42982 (16)	0.57709 (11)	0.0182 (2)
C2	0.36053 (17)	0.57316 (16)	0.67248 (12)	0.0181 (2)
C3	0.41614 (17)	0.58856 (17)	0.79952 (11)	0.0199 (2)
H3	0.3880	0.4888	0.8433	0.024*
C4	0.51439 (18)	0.75637 (17)	0.85853 (12)	0.0210 (3)
C5	0.55869 (18)	0.90590 (17)	0.79684 (12)	0.0224 (3)
H5	0.6274	1.0181	0.8415	0.027*
C6	0.50280 (18)	0.89127 (17)	0.67078 (12)	0.0219 (3)
H6	0.5305	0.9910	0.6270	0.026*
C7	0.40473 (17)	0.72374 (16)	0.61254 (11)	0.0184 (2)
C8	0.24675 (17)	0.50143 (16)	0.46942 (12)	0.0184 (2)
C9	0.16187 (17)	0.42847 (17)	0.34289 (11)	0.0185 (2)
C10	0.01060 (18)	0.26868 (18)	0.32117 (12)	0.0221 (3)
H10	−0.0400	0.2079	0.3902	0.026*
C11	−0.06688 (19)	0.19735 (18)	0.20057 (13)	0.0247 (3)
H11	−0.1679	0.0873	0.1854	0.030*
C12	0.00739 (19)	0.29133 (19)	0.10332 (12)	0.0244 (3)
C13	0.15404 (19)	0.45109 (18)	0.12037 (12)	0.0242 (3)
H13	0.2003	0.5126	0.0508	0.029*
C14	0.23250 (18)	0.52006 (17)	0.24092 (12)	0.0209 (3)
H14	0.3344	0.6296	0.2547	0.025*
C15	−0.01001 (19)	0.18735 (19)	0.68205 (14)	0.0275 (3)
H15A	−0.0655	0.0691	0.7067	0.041*
H15B	−0.1017	0.2104	0.6304	0.041*
H15C	0.0307	0.2731	0.7574	0.041*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.02235 (17)	0.01534 (16)	0.02005 (16)	0.00628 (12)	0.00176 (12)	0.00143 (11)
Cl	0.03404 (19)	0.02564 (18)	0.01697 (16)	0.00867 (14)	−0.00482 (12)	−0.00284 (12)
F	0.0440 (5)	0.0401 (5)	0.0167 (4)	0.0061 (4)	−0.0093 (4)	−0.0020 (4)
O1	0.0235 (4)	0.0174 (4)	0.0166 (4)	0.0059 (4)	−0.0014 (3)	0.0018 (3)
O2	0.0262 (5)	0.0258 (5)	0.0373 (6)	0.0108 (4)	−0.0019 (4)	0.0103 (4)
C1	0.0202 (6)	0.0162 (6)	0.0170 (5)	0.0060 (5)	0.0003 (4)	0.0007 (4)
C2	0.0187 (6)	0.0161 (6)	0.0190 (6)	0.0067 (5)	0.0007 (4)	0.0008 (4)
C3	0.0230 (6)	0.0189 (6)	0.0174 (6)	0.0081 (5)	−0.0001 (5)	0.0012 (5)
C4	0.0219 (6)	0.0232 (6)	0.0174 (6)	0.0094 (5)	−0.0014 (5)	−0.0012 (5)
C5	0.0227 (6)	0.0184 (6)	0.0227 (6)	0.0057 (5)	−0.0009 (5)	−0.0024 (5)
C6	0.0235 (6)	0.0171 (6)	0.0234 (6)	0.0060 (5)	0.0007 (5)	0.0026 (5)
C7	0.0191 (6)	0.0197 (6)	0.0160 (5)	0.0076 (5)	−0.0005 (4)	0.0013 (4)
C8	0.0179 (6)	0.0169 (6)	0.0189 (6)	0.0054 (5)	0.0011 (4)	0.0009 (4)
C9	0.0194 (6)	0.0209 (6)	0.0161 (5)	0.0093 (5)	−0.0002 (4)	0.0013 (4)
C10	0.0216 (6)	0.0240 (6)	0.0187 (6)	0.0064 (5)	0.0002 (5)	0.0041 (5)
C11	0.0222 (6)	0.0243 (6)	0.0231 (6)	0.0050 (5)	−0.0029 (5)	0.0006 (5)
C12	0.0272 (7)	0.0296 (7)	0.0150 (6)	0.0111 (5)	−0.0048 (5)	−0.0018 (5)
C13	0.0275 (7)	0.0281 (7)	0.0175 (6)	0.0108 (5)	0.0020 (5)	0.0051 (5)
C14	0.0214 (6)	0.0207 (6)	0.0199 (6)	0.0073 (5)	0.0010 (5)	0.0028 (5)
C15	0.0248 (7)	0.0276 (7)	0.0317 (7)	0.0100 (5)	0.0082 (5)	0.0087 (6)

Geometric parameters (Å, °)

S—O2	1.490 (1)	C6—C7	1.380 (2)
S—C1	1.766 (1)	C6—H6	0.9500
S—C15	1.792 (1)	C8—C9	1.458 (2)
Cl—C4	1.742 (1)	C9—C10	1.396 (2)
Cl—O2^{i}	3.244 (1)	C9—C14	1.404 (2)
F—C12	1.358 (1)	C10—C11	1.385 (2)
O1—C7	1.376 (2)	C10—H10	0.9500
O1—C8	1.378 (2)	C11—C12	1.377 (2)
C1—C8	1.368 (2)	C11—H11	0.9500
C1—C2	1.444 (2)	C12—C13	1.377 (2)
C2—C7	1.395 (2)	C13—C14	1.383 (2)
C2—C3	1.396 (2)	C13—H13	0.9500
C3—C4	1.385 (2)	C14—H14	0.9500
C3—H3	0.9500	C15—H15A	0.9800
C4—C5	1.398 (2)	C15—H15B	0.9800
C5—C6	1.387 (2)	C15—H15C	0.9800
C5—H5	0.9500		
O2—S—C1	107.25 (6)	C1—C8—C9	133.83 (12)
O2—S—C15	106.04 (7)	O1—C8—C9	115.54 (11)
C4—Cl—O2^{i}	174.59 (5)	C10—C9—C14	119.21 (11)
C1—S—C15	97.55 (6)	C10—C9—C8	121.09 (11)

C7—O1—C8	106.71 (9)	C14—C9—C8	119.70 (11)
C8—C1—C2	106.97 (11)	C11—C10—C9	121.01 (12)
C8—C1—S	126.78 (10)	C11—C10—H10	119.5
C2—C1—S	126.01 (9)	C9—C10—H10	119.5
C7—C2—C3	119.43 (11)	C12—C11—C10	117.77 (12)
C7—C2—C1	105.20 (11)	C12—C11—H11	121.1
C3—C2—C1	135.37 (12)	C10—C11—H11	121.1
C4—C3—C2	116.72 (12)	F—C12—C11	118.33 (12)
C4—C3—H3	121.6	F—C12—C13	118.37 (12)
C2—C3—H3	121.6	C11—C12—C13	123.30 (12)
C3—C4—C5	123.13 (12)	C12—C13—C14	118.55 (12)
C3—C4—Cl	118.45 (10)	C12—C13—H13	120.7
C5—C4—Cl	118.42 (10)	C14—C13—H13	120.7
C6—C5—C4	120.34 (12)	C13—C14—C9	120.13 (12)
C6—C5—H5	119.8	C13—C14—H14	119.9
C4—C5—H5	119.8	C9—C14—H14	119.9
C7—C6—C5	116.26 (12)	S—C15—H15A	109.5
C7—C6—H6	121.9	S—C15—H15B	109.5
C5—C6—H6	121.9	H15A—C15—H15B	109.5
O1—C7—C6	125.40 (11)	S—C15—H15C	109.5
O1—C7—C2	110.47 (11)	H15A—C15—H15C	109.5
C6—C7—C2	124.12 (12)	H15B—C15—H15C	109.5
C1—C8—O1	110.63 (11)		

O2—S—C1—C8	141.61 (12)	C1—C2—C7—C6	−179.94 (12)
C15—S—C1—C8	−108.94 (12)	C2—C1—C8—O1	0.03 (14)
O2—S—C1—C2	−31.99 (12)	S—C1—C8—O1	−174.55 (9)
C15—S—C1—C2	77.46 (12)	C2—C1—C8—C9	−179.35 (13)
C8—C1—C2—C7	−0.78 (13)	S—C1—C8—C9	6.1 (2)
S—C1—C2—C7	173.85 (9)	C7—O1—C8—C1	0.75 (13)
C8—C1—C2—C3	178.83 (14)	C7—O1—C8—C9	−179.75 (10)
S—C1—C2—C3	−6.5 (2)	C1—C8—C9—C10	26.5 (2)
C7—C2—C3—C4	−0.26 (18)	O1—C8—C9—C10	−152.84 (12)
C1—C2—C3—C4	−179.83 (13)	C1—C8—C9—C14	−153.67 (14)
C2—C3—C4—C5	−0.19 (19)	O1—C8—C9—C14	26.98 (16)
C2—C3—C4—Cl	179.52 (9)	C14—C9—C10—C11	1.87 (19)
C3—C4—C5—C6	0.6 (2)	C8—C9—C10—C11	−178.31 (12)
Cl—C4—C5—C6	−179.15 (10)	C9—C10—C11—C12	−1.6 (2)
C4—C5—C6—C7	−0.44 (19)	C10—C11—C12—F	−179.67 (12)
C8—O1—C7—C6	179.96 (12)	C10—C11—C12—C13	0.2 (2)
C8—O1—C7—C2	−1.27 (13)	F—C12—C13—C14	−179.31 (12)
C5—C6—C7—O1	178.59 (11)	C11—C12—C13—C14	0.8 (2)
C5—C6—C7—C2	−0.01 (19)	C12—C13—C14—C9	−0.49 (19)
C3—C2—C7—O1	−178.41 (11)	C10—C9—C14—C13	−0.81 (19)
C1—C2—C7—O1	1.27 (13)	C8—C9—C14—C13	179.37 (12)
C3—C2—C7—C6	0.37 (19)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14···O2^{ii}	0.95	2.56	3.4287 (16)	152

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

Fig. 1

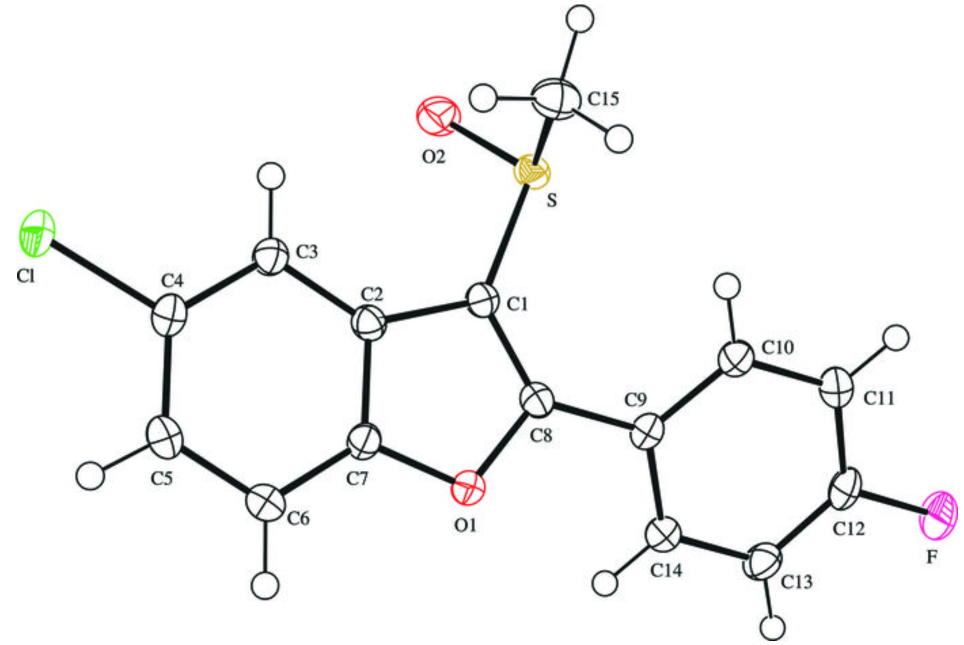
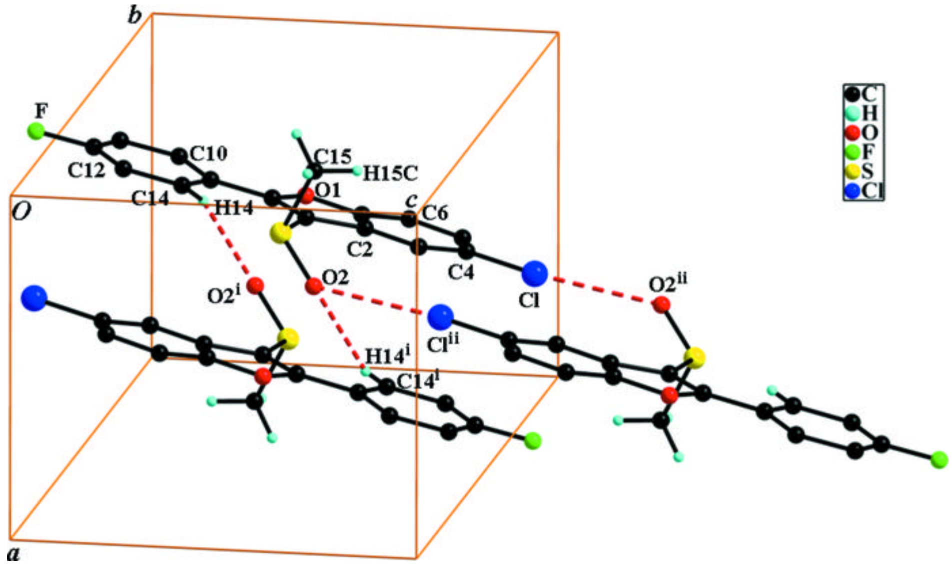


Fig. 2


b
F
C10
C12
C15
H15C
C14
O
H14
O1
c
C6
C2
C4
O2
Cl
O2i
O2ii
Clii
H14i
C14i
a
C
H
O
F
S
Cl