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5-Fluoro-2-(2-fluorophenyl)-3-methylsulfinyl-1-benzofuran

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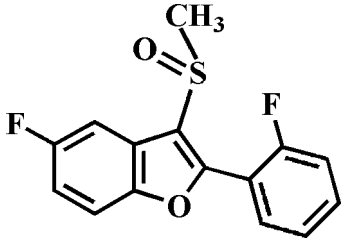
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.100; data-to-parameter ratio = 17.1.

In the title compound, $C_{15}H_{10}F_2O_2S$, the dihedral angle between the plane of the benzofuran ring system (r.m.s. deviation = 0.015 Å) and that of the 2-fluorophenyl ring is 28.53 (6)°. In the crystal, molecules are linked by C—H···O and C—H···F hydrogen bonds, and by π–π interactions between the furan and benzene rings of neighbouring molecules [centroid–centroid distance = 3.625 (2) Å], forming a three-dimensional network.

Related literature

For background information and the crystal structures of related compounds, see: Choi *et al.* (2009*a*,*b*, 2012).



Experimental

Crystal data

$C_{15}H_{10}F_2O_2S$
M_r = 292.29
Monoclinic, $P2_1/c$
a = 8.6184 (2) Å
b = 16.8358 (4) Å
c = 9.4019 (2) Å
β = 111.254 (1)°
V = 1271.40 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 173 K
0.55 × 0.27 × 0.24 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.863, T_{max} = 0.937
12086 measured reflections
3118 independent reflections
2650 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.100
S = 1.07
3118 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O2^{i}	0.95	2.52	3.343 (2)	145
C12—H12···O2^{ii}	0.95	2.39	3.326 (2)	166
C15—H15*A*···F1^{iii}	0.98	2.54	3.419 (2)	149

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

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 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *SHELXL97*.

Supporting information for this paper is available from the IUCr electronic archives (Reference: BT6985).

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supporting information

Acta Cryst. (2014). E**70**, o829 [https://doi.org/10.1107/S1600536814014810]

5-Fluoro-2-(2-fluorophenyl)-3-methylsulfinyl-1-benzofuran

Hong Dae Choi, Pil Ja Seo and Uk Lee

S1. Comment

As a part of our ongoing project of 5-fluoro-3-methylsulfinyl-1-benzofuran derivatives containing 4-bromophenyl (Choi *et al.*, 2009*a*), 4-fluorophenyl (Choi *et al.*, 2009*b*) and 4-methylphenyl (Choi *et al.*, 2012) substituents in 2-position, 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 0.015 (1) Å from the least-squares plane defined by the nine constituent atoms. The 2-fluorophenyl ring is essentially planar, with a mean deviation of 0.053 (1) Å from the least-squares plane defined by the six constituent atoms. The dihedral angle formed by the benzofuran ring system and the 2-fluorophenyl ring is 28.53 (6)°. In the crystal structure (Fig. 2), molecules are inked by C—H···O and C—H···F hydrogen bonds (Table 1), and by π–π interactions between the furan and benzene rings of neighbouring molecules, with a Cg1···Cg2^{iv} distance of 3.625 (2) Å and an interplanar distance of 3.295 (2) Å resulting in a slippage of 1.511 (2) Å (Cg1 and Cg2 are the centroids of the C1/C2/C7/O1/C8 furan ring and the C2–C7 benzene ring, respectively), forming a three-dimensional network.

S2. Experimental

3-Chloroperoxybenzoic acid (77%, 269 mg, 1.2 mmol) was added in small portions to a stirred solution of 5-fluoro-2-(2-fluorophenyl)-3-methylsulfanyl-1-benzofuran (304 mg, 1.1 mmol) in dichloromethane (35 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. 412–413 K; R_f = 0.46 (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 using the SHELXL-97 command AFIX 137 (Sheldrick, 2008).

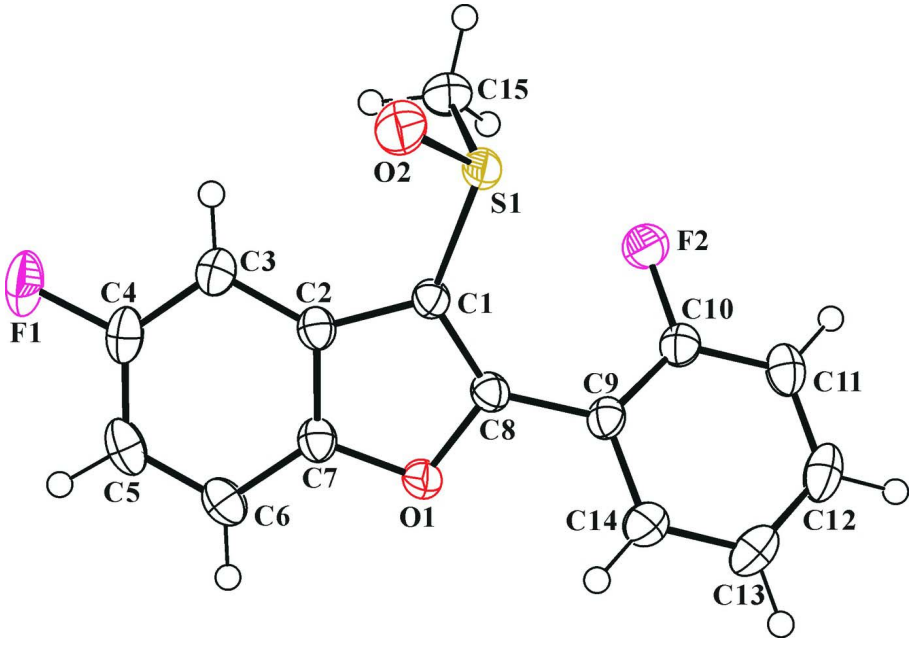

Figure 1

The molecular structure of the title molecule with the atom numbering scheme The displacement ellipsoids are drawn at the 50% probability level. The hydrogen atoms are presented as small spheres of arbitrary radius.

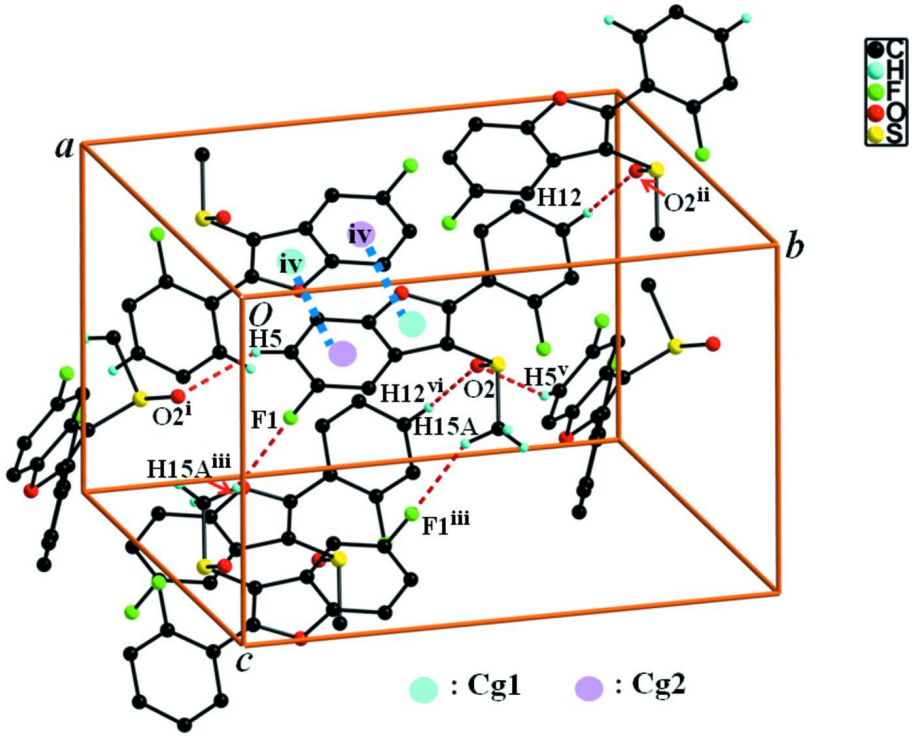

Figure 2

A view of the C—H···O, C—H···F and π–π interactions (dotted lines) in the crystal structure of the title compound. H atoms non-participating in hydrogen-bonding were omitted for clarity. [Symmetry codes: (i) - x + 2, y - 1/2, - z + 3/2; (ii) x - 1, y, z -1; (iii) - x + 2, - y + 1, - z + 2; (iv) - x + 2, - y + 1, - z + 1; (v) - x + 1, y + 1/2, - z + 3/2; (vi) x + 1, y, z + 1.]

5-Fluoro-2-(2-fluorophenyl)-3-methylsulfinyl-1-benzofuran

Crystal data

$C_{15}H_{10}F_2O_2S$
$M_r = 292.29$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.6184$ (2) Å
$b = 16.8358$ (4) Å
$c = 9.4019$ (2) Å
$\beta = 111.254$ (1)°
$V = 1271.40$ (5) Å^3
$Z = 4$
$F(000) = 600$
$D_x = 1.527$ Mg m^{-3}
Melting point = 413–412 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4357 reflections
$\theta = 2.4$–27.8°
$\mu = 0.28$ mm^{-1}
$T = 173$ K
Block, colourless
0.55 × 0.27 × 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)
$T_{min} = 0.863$, $T_{max} = 0.937$
12086 measured reflections
3118 independent reflections
2650 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.4°$
$h = -10 \rightarrow 11$
$k = -22 \rightarrow 19$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.100$
$S = 1.07$
3118 reflections
182 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
$w = 1/[\sigma^2(F_o^2) + (0.0492P)^2 + 0.3389P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.40$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.71915 (5)	0.69054 (2)	0.61445 (4)	0.02927 (12)
F1	1.13845 (13)	0.42819 (7)	0.91201 (11)	0.0485 (3)
F2	0.38183 (11)	0.67630 (6)	0.41827 (10)	0.0374 (2)

O1	0.67546 (12)	0.49850 (6)	0.36067 (11)	0.0281 (2)
O2	0.89953 (15)	0.70691 (7)	0.69905 (14)	0.0431 (3)
C1	0.71116 (17)	0.59444 (8)	0.53646 (16)	0.0234 (3)
C2	0.82307 (17)	0.53035 (9)	0.60821 (17)	0.0250 (3)
C3	0.94153 (18)	0.51622 (10)	0.75344 (17)	0.0299 (3)
H3	0.9632	0.5534	0.8344	0.036*
C4	1.02416 (19)	0.44539 (10)	0.77150 (18)	0.0336 (4)
C5	1.0016 (2)	0.38969 (10)	0.6577 (2)	0.0361 (4)
H5	1.0650	0.3421	0.6785	0.043*
C6	0.8859 (2)	0.40384 (9)	0.5134 (2)	0.0330 (3)
H6	0.8681	0.3673	0.4320	0.040*
C7	0.79767 (17)	0.47404 (9)	0.49421 (17)	0.0267 (3)
C8	0.62565 (17)	0.57246 (9)	0.38947 (16)	0.0242 (3)
C9	0.49420 (17)	0.60820 (9)	0.25958 (16)	0.0254 (3)
C10	0.37363 (18)	0.65773 (9)	0.27581 (17)	0.0271 (3)
C11	0.24304 (19)	0.68798 (10)	0.15475 (19)	0.0324 (3)
H11	0.1626	0.7215	0.1713	0.039*
C12	0.2317 (2)	0.66846 (11)	0.00904 (19)	0.0382 (4)
H12	0.1426	0.6886	−0.0764	0.046*
C13	0.3500 (2)	0.61954 (12)	−0.01285 (19)	0.0417 (4)
H13	0.3418	0.6064	−0.1136	0.050*
C14	0.4797 (2)	0.58969 (11)	0.10995 (18)	0.0351 (4)
H14	0.5601	0.5562	0.0930	0.042*
C15	0.6325 (2)	0.66632 (12)	0.7563 (2)	0.0435 (4)
H15A	0.6984	0.6239	0.8221	0.065*
H15B	0.5173	0.6484	0.7060	0.065*
H15C	0.6347	0.7135	0.8181	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0313 (2)	0.0205 (2)	0.0279 (2)	0.00149 (14)	0.00106 (15)	−0.00264 (14)
F1	0.0421 (6)	0.0544 (7)	0.0399 (6)	0.0162 (5)	0.0039 (5)	0.0204 (5)
F2	0.0311 (5)	0.0469 (6)	0.0322 (5)	0.0074 (4)	0.0092 (4)	−0.0046 (4)
O1	0.0285 (5)	0.0232 (5)	0.0278 (5)	0.0023 (4)	0.0045 (4)	−0.0042 (4)
O2	0.0356 (6)	0.0315 (7)	0.0463 (7)	−0.0063 (5)	−0.0041 (5)	−0.0079 (5)
C1	0.0240 (7)	0.0200 (7)	0.0239 (7)	−0.0001 (5)	0.0060 (5)	0.0002 (5)
C2	0.0239 (7)	0.0225 (7)	0.0278 (7)	0.0003 (5)	0.0085 (6)	0.0026 (6)
C3	0.0289 (7)	0.0308 (8)	0.0271 (8)	0.0014 (6)	0.0068 (6)	0.0047 (6)
C4	0.0273 (7)	0.0373 (9)	0.0332 (8)	0.0043 (6)	0.0073 (6)	0.0148 (7)
C5	0.0333 (8)	0.0262 (8)	0.0509 (10)	0.0080 (6)	0.0177 (7)	0.0123 (7)
C6	0.0354 (8)	0.0223 (8)	0.0426 (9)	0.0026 (6)	0.0155 (7)	0.0013 (7)
C7	0.0256 (7)	0.0234 (7)	0.0293 (8)	0.0004 (6)	0.0079 (6)	0.0026 (6)
C8	0.0233 (7)	0.0207 (7)	0.0269 (7)	−0.0001 (5)	0.0071 (6)	−0.0013 (6)
C9	0.0229 (7)	0.0235 (7)	0.0251 (7)	−0.0021 (5)	0.0029 (5)	−0.0002 (6)
C10	0.0250 (7)	0.0253 (7)	0.0277 (7)	−0.0033 (6)	0.0056 (6)	−0.0001 (6)
C11	0.0247 (7)	0.0280 (8)	0.0398 (9)	0.0000 (6)	0.0059 (6)	0.0058 (7)
C12	0.0305 (8)	0.0389 (10)	0.0333 (9)	−0.0031 (7)	−0.0027 (7)	0.0103 (7)

C13	0.0409 (9)	0.0522 (11)	0.0237 (8)	−0.0017 (8)	0.0020 (7)	0.0002 (7)
C14	0.0332 (8)	0.0390 (10)	0.0286 (8)	0.0031 (7)	0.0059 (6)	−0.0041 (7)
C15	0.0510 (11)	0.0494 (11)	0.0300 (9)	0.0126 (9)	0.0143 (8)	−0.0051 (8)

Geometric parameters (Å, º)

S1—O2	1.4930 (12)	C6—C7	1.381 (2)
S1—C1	1.7675 (14)	C6—H6	0.9500
S1—C15	1.7952 (18)	C8—C9	1.461 (2)
F1—C4	1.3623 (18)	C9—C10	1.383 (2)
F2—C10	1.3521 (17)	C9—C14	1.402 (2)
O1—C8	1.3757 (17)	C10—C11	1.376 (2)
O1—C7	1.3772 (18)	C11—C12	1.377 (2)
C1—C8	1.3606 (19)	C11—H11	0.9500
C1—C2	1.441 (2)	C12—C13	1.383 (3)
C2—C7	1.388 (2)	C12—H12	0.9500
C2—C3	1.397 (2)	C13—C14	1.378 (2)
C3—C4	1.368 (2)	C13—H13	0.9500
C3—H3	0.9500	C14—H14	0.9500
C4—C5	1.383 (3)	C15—H15A	0.9800
C5—C6	1.382 (2)	C15—H15B	0.9800
C5—H5	0.9500	C15—H15C	0.9800
O2—S1—C1	105.45 (7)	C1—C8—C9	135.03 (14)
O2—S1—C15	106.22 (8)	O1—C8—C9	114.27 (12)
C1—S1—C15	97.82 (8)	C10—C9—C14	116.59 (14)
C8—O1—C7	106.33 (11)	C10—C9—C8	122.83 (13)
C8—C1—C2	107.22 (13)	C14—C9—C8	120.49 (14)
C8—C1—S1	126.55 (11)	F2—C10—C11	117.86 (14)
C2—C1—S1	124.91 (11)	F2—C10—C9	118.49 (13)
C7—C2—C3	119.55 (14)	C11—C10—C9	123.63 (14)
C7—C2—C1	104.98 (13)	C10—C11—C12	118.45 (15)
C3—C2—C1	135.45 (14)	C10—C11—H11	120.8
C4—C3—C2	115.65 (15)	C12—C11—H11	120.8
C4—C3—H3	122.2	C11—C12—C13	119.99 (15)
C2—C3—H3	122.2	C11—C12—H12	120.0
F1—C4—C3	117.82 (15)	C13—C12—H12	120.0
F1—C4—C5	117.12 (14)	C14—C13—C12	120.69 (16)
C3—C4—C5	125.06 (15)	C14—C13—H13	119.7
C6—C5—C4	119.46 (15)	C12—C13—H13	119.7
C6—C5—H5	120.3	C13—C14—C9	120.65 (16)
C4—C5—H5	120.3	C13—C14—H14	119.7
C7—C6—C5	116.30 (15)	C9—C14—H14	119.7
C7—C6—H6	121.9	S1—C15—H15A	109.5
C5—C6—H6	121.9	S1—C15—H15B	109.5
O1—C7—C6	125.31 (14)	H15A—C15—H15B	109.5
O1—C7—C2	110.75 (12)	S1—C15—H15C	109.5
C6—C7—C2	123.93 (14)	H15A—C15—H15C	109.5

C1—C8—O1	110.69 (12)	H15B—C15—H15C	109.5
O2—S1—C1—C8	−131.06 (13)	C1—C2—C7—C6	177.33 (14)
C15—S1—C1—C8	119.65 (14)	C2—C1—C8—O1	−0.41 (16)
O2—S1—C1—C2	34.10 (14)	S1—C1—C8—O1	166.89 (10)
C15—S1—C1—C2	−75.19 (14)	C2—C1—C8—C9	178.28 (15)
C8—C1—C2—C7	1.30 (15)	S1—C1—C8—C9	−14.4 (2)
S1—C1—C2—C7	−166.26 (11)	C7—O1—C8—C1	−0.67 (15)
C8—C1—C2—C3	179.85 (16)	C7—O1—C8—C9	−179.65 (12)
S1—C1—C2—C3	12.3 (2)	C1—C8—C9—C10	−31.3 (3)
C7—C2—C3—C4	−0.5 (2)	O1—C8—C9—C10	147.36 (14)
C1—C2—C3—C4	−178.85 (16)	C1—C8—C9—C14	152.31 (17)
C2—C3—C4—F1	−178.67 (13)	O1—C8—C9—C14	−29.0 (2)
C2—C3—C4—C5	1.7 (2)	C14—C9—C10—F2	179.21 (13)
F1—C4—C5—C6	179.35 (14)	C8—C9—C10—F2	2.7 (2)
C3—C4—C5—C6	−1.0 (3)	C14—C9—C10—C11	0.7 (2)
C4—C5—C6—C7	−0.9 (2)	C8—C9—C10—C11	−175.83 (14)
C8—O1—C7—C6	−177.54 (14)	F2—C10—C11—C12	−178.95 (14)
C8—O1—C7—C2	1.55 (16)	C9—C10—C11—C12	−0.4 (2)
C5—C6—C7—O1	−178.87 (14)	C10—C11—C12—C13	0.0 (3)
C5—C6—C7—C2	2.2 (2)	C11—C12—C13—C14	0.2 (3)
C3—C2—C7—O1	179.41 (12)	C12—C13—C14—C9	0.1 (3)
C1—C2—C7—O1	−1.76 (16)	C10—C9—C14—C13	−0.5 (2)
C3—C2—C7—C6	−1.5 (2)	C8—C9—C14—C13	176.09 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O2^{i}	0.95	2.52	3.343 (2)	145
C12—H12···O2^{ii}	0.95	2.39	3.326 (2)	166
C15—H15*A*···F1^{iii}	0.98	2.54	3.419 (2)	149

Symmetry codes: (i) −*x*+2, *y*−1/2, −*z*+3/2; (ii) *x*−1, *y*, *z*−1; (iii) −*x*+2, −*y*+1, −*z*+2.