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3-(4-Fluorophenylsulfinyl)-5-iodo-2,7-dimethyl-1-benzofuran

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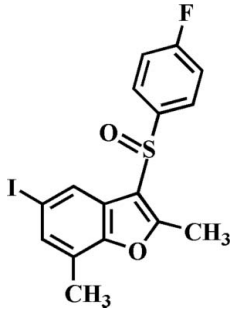
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.025; wR factor = 0.065; data-to-parameter ratio = 18.1.

In the title compound, $C_{16}H_{12}FIO_2S$, the 4-fluorophenyl ring makes a dihedral angle of 80.21 (6)° with the mean plane of the benzofuran fragment. In the crystal, molecules are linked through weak intermolecular C—H···O hydrogen bonds. The crystal structure also exhibits an intermolecular I···F contact [3.423 (2) Å].

Related literature

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 our previous structural studies of related 3-(4-fluorophenylsulfinyl)-5-halo-2-methyl-1-benzofuran derivatives, see: Choi *et al.* (2010*a*,*b*,*c*).



Experimental

Crystal data

$C_{16}H_{12}FIO_2S$ M_r = 414.22
Triclinic, $P\overline{1}$
a = 9.0845 (2) Å
b = 9.2761 (2) Å
c = 10.1252 (2) Å
α = 71.315 (1)°
β = 80.838 (1)°
γ = 70.485 (1)°
V = 760.53 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.25 mm^{-1}
T = 173 K
0.25 × 0.23 × 0.20 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.603, T_{max} = 0.666
13286 measured reflections
3477 independent reflections
3276 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.065
S = 1.15
3477 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −1.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O2^{i}	0.95	2.51	3.444 (3)	169
C9—H9A···O2^{ii}	0.98	2.48	3.437 (3)	165
C15—H15···O1^{iii}	0.95	2.49	3.342 (3)	149

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

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: BV2179).

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supporting information

Acta Cryst. (2011). E**67**, o1082 [doi:10.1107/S1600536811012591]

3-(4-Fluorophenylsulfinyl)-5-iodo-2,7-dimethyl-1-benzofuran

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

S1. Comment

A series of benzofuran ring system have attracted much attention owing to their interesting pharmacological properties such as antifungal, antitumor and antiviral, antimicrobial activities (Aslam *et al.*, 2006, Galal *et al.*, 2009, Khan *et al.*, 2005). These compounds widely occur in nature (Akgul & Anil, 2003; Soekamto *et al.*, 2003). As a part of our study of the substituent effect on the solid state structures of 3-(4-fluorophenylsulfinyl)-5-halo-2-methyl-1-benzofuran analogues (Choi *et al.*, 2010*a,b,c*), we report here 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 0.009 (2) Å from the least-squares plane defined by the nine constituent atoms. The dihedral angle formed by the mean plane of the benzofuran fragment and the 4-fluorophenyl ring is 80.21 (6)°. The molecular packing (Fig. 2) is stabilized by weak intermolecular C—H···O hydrogen bonds; the first one between a benzene H atom and the oxygen of the S═O unit (Table 1: C3—H3···O2^{i}), and the second one between a methyl H atom and the oxygen of the S═O unit (Table 1: C9—H9A···O2^{ii}), and the third one between a 4-fluorophenyl H atom and the furan O atom (Table 1; C15—H15···O1^{iii}). The crystal packing (Fig. 2) is further stabilized by an I···F contact at 3.423 (2) Å [C4—I1···F1^{iv} = 161.10 (6) °].

S2. Experimental

77% 3-chloroperoxybenzoic acid (202 mg, 0.9 mmol) was added in small portions to a stirred solution of 3-(4-fluorophenylsulfanyl)-5-iodo-2,7-dimethyl-1-benzofuran (318 mg, 0.8 mmol) in dichloromethane (40 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 72%, m.p. 423–424 K; Rf = 0.65 (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. U_{iso}(H) =1.2U_{eq}(C) for aryl and 1.5Ueq(C) for methyl H atoms.

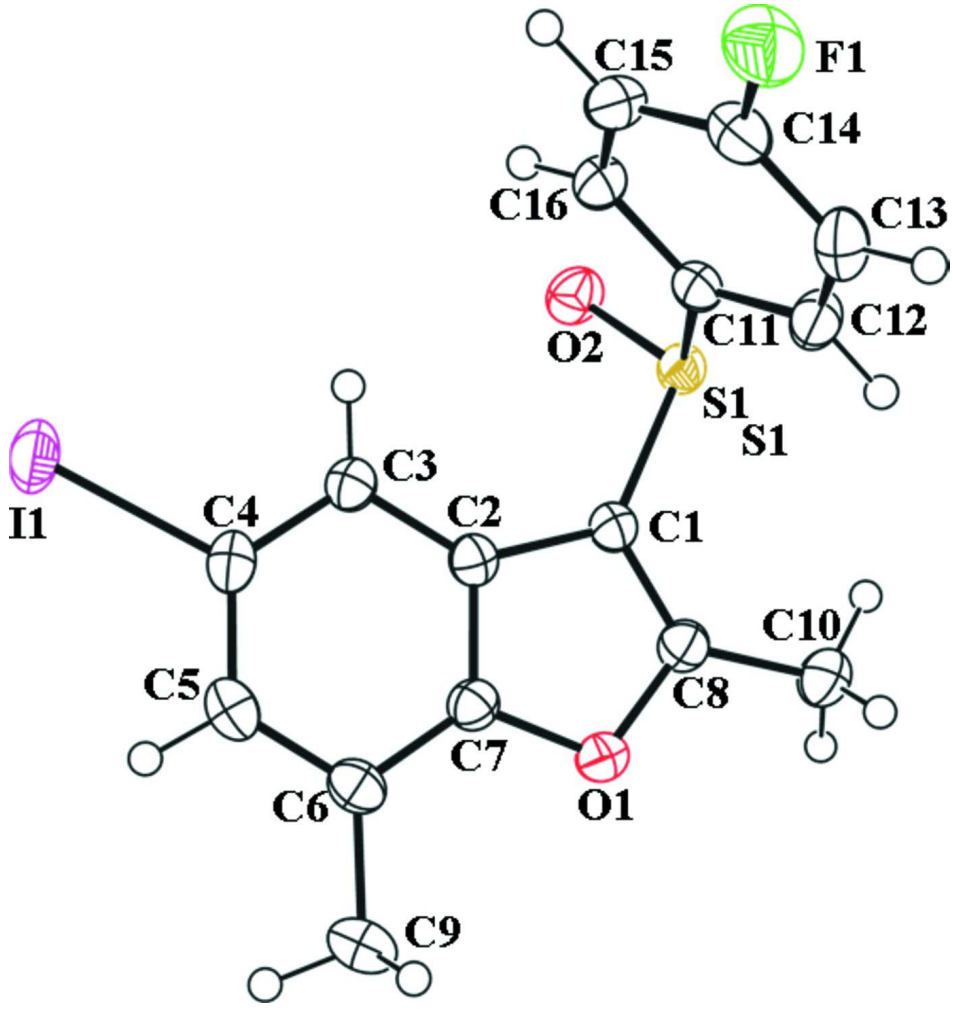

Figure 1

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.

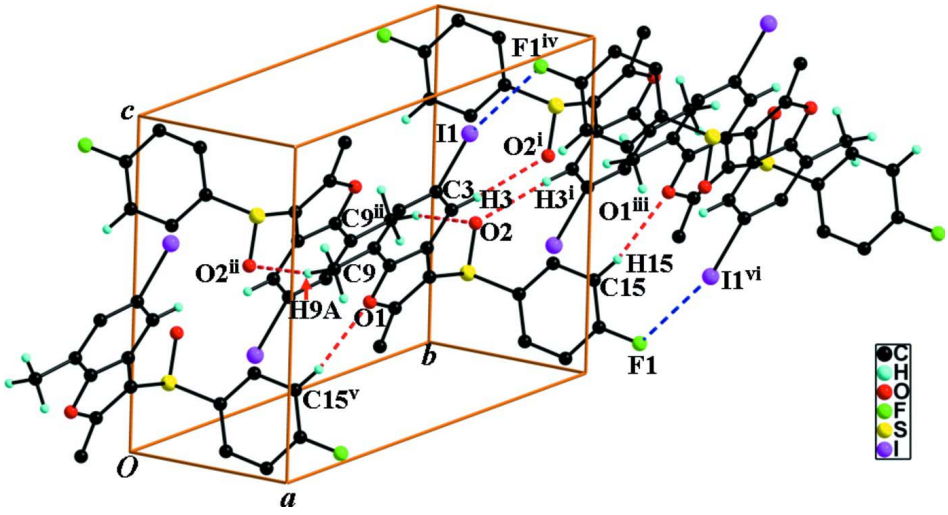

Figure 2

A view of the C—H···O and I···F interactions (dotted lines) in the crystal structure of the title compound. [Symmetry codes: (i) - *x* + 1, - *y* + 2, - *z* + 1 ; (ii) - *x* + 1, - *y* + 1, - *z* + 1; (iii) *x*, *y* + 1, *z*; (iv) - *x* + 2, - *y* + 2, - *z* + 1; (v) *x*, *y* - 1, *z*; (vi) - *x* + 2, - *y* + 2, - *z* + 1.]

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Crystal data

$C_{16}H_{12}FIO_2S$
$M_r = 414.22$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 9.0845$ (2) Å
$b = 9.2761$ (2) Å
$c = 10.1252$ (2) Å
$\alpha = 71.315$ (1)°
$\beta = 80.838$ (1)°
$\gamma = 70.485$ (1)°
$V = 760.53$ (3) Å^3
$Z = 2$
$F(000) = 404$
$D_x = 1.809$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9933 reflections
$\theta = 2.4$–27.5°
$\mu = 2.25$ mm^{-1}
$T = 173$ K
Block, colourless
0.25 × 0.23 × 0.20 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)
$T_{min} = 0.603$, $T_{max} = 0.666$
13286 measured reflections
3477 independent reflections
3276 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.1°$
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 11$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.065$
$S = 1.15$
3477 reflections
192 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

$w = 1/[\sigma^2(F_o^2) + (0.0297P)^2 + 0.3709P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -1.25$ 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 $F^2 > 2\text{sigma}(F^2)$ 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}
I1	0.885022 (19)	0.64773 (2)	0.817121 (15)	0.04100 (7)
S1	0.46511 (6)	0.87701 (6)	0.28833 (5)	0.02623 (11)
F1	0.91534 (19)	1.2241 (2)	0.00671 (16)	0.0487 (4)
O1	0.70380 (17)	0.42169 (18)	0.37299 (15)	0.0282 (3)
O2	0.39368 (18)	0.9370 (2)	0.41076 (16)	0.0337 (3)
C1	0.5892 (2)	0.6822 (2)	0.3520 (2)	0.0247 (4)
C2	0.6891 (2)	0.6179 (2)	0.4686 (2)	0.0242 (4)
C3	0.7247 (2)	0.6783 (3)	0.5651 (2)	0.0258 (4)
H3	0.6807	0.7873	0.5631	0.031*
C4	0.8280 (2)	0.5699 (3)	0.6641 (2)	0.0289 (4)
C5	0.8967 (2)	0.4099 (3)	0.6678 (2)	0.0302 (4)
H5	0.9687	0.3419	0.7369	0.036*
C6	0.8620 (2)	0.3475 (3)	0.5724 (2)	0.0283 (4)
C8	0.6016 (2)	0.5613 (3)	0.3001 (2)	0.0269 (4)
C7	0.7563 (2)	0.4570 (3)	0.4756 (2)	0.0255 (4)
C9	0.9340 (3)	0.1760 (3)	0.5733 (3)	0.0369 (5)
H9A	0.8521	0.1323	0.5663	0.055*
H9B	0.9865	0.1146	0.6604	0.055*
H9C	1.0105	0.1694	0.4937	0.055*
C10	0.5306 (3)	0.5520 (3)	0.1823 (3)	0.0362 (5)
H10A	0.4481	0.6521	0.1470	0.054*
H10B	0.4857	0.4632	0.2145	0.054*
H10C	0.6112	0.5344	0.1074	0.054*
C11	0.6111 (2)	0.9745 (2)	0.2075 (2)	0.0254 (4)
C12	0.6769 (3)	0.9632 (3)	0.0763 (2)	0.0313 (5)
H12	0.6505	0.8982	0.0337	0.038*
C13	0.7807 (3)	1.0468 (3)	0.0079 (2)	0.0353 (5)
H13	0.8270	1.0405	−0.0819	0.042*
C14	0.8148 (3)	1.1395 (3)	0.0736 (2)	0.0333 (5)
C15	0.7536 (3)	1.1507 (3)	0.2047 (2)	0.0328 (5)

H15	0.7828	1.2132	0.2481	0.039*
C16	0.6476 (3)	1.0676 (3)	0.2718 (2)	0.0296 (4)
H16	0.6007	1.0750	0.3612	0.036*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.05179 (11)	0.04985 (12)	0.02844 (9)	−0.02072 (8)	−0.01085 (7)	−0.01101 (7)
S1	0.0251 (2)	0.0264 (2)	0.0265 (2)	−0.0037 (2)	−0.00507 (18)	−0.0092 (2)
F1	0.0549 (9)	0.0500 (9)	0.0437 (8)	−0.0300 (8)	0.0008 (7)	−0.0036 (7)
O1	0.0311 (7)	0.0245 (7)	0.0302 (7)	−0.0066 (6)	−0.0046 (6)	−0.0101 (6)
O2	0.0319 (8)	0.0362 (9)	0.0336 (8)	−0.0068 (7)	0.0023 (6)	−0.0164 (7)
C1	0.0253 (9)	0.0243 (10)	0.0237 (9)	−0.0055 (8)	−0.0033 (7)	−0.0070 (8)
C2	0.0230 (9)	0.0262 (10)	0.0228 (9)	−0.0072 (8)	−0.0007 (7)	−0.0066 (8)
C3	0.0264 (9)	0.0276 (10)	0.0246 (9)	−0.0094 (8)	−0.0008 (7)	−0.0081 (8)
C4	0.0311 (10)	0.0363 (12)	0.0225 (9)	−0.0143 (9)	−0.0024 (8)	−0.0080 (9)
C5	0.0277 (10)	0.0325 (11)	0.0262 (10)	−0.0096 (9)	−0.0040 (8)	−0.0009 (9)
C6	0.0260 (9)	0.0261 (10)	0.0292 (10)	−0.0080 (8)	−0.0004 (8)	−0.0037 (8)
C8	0.0267 (9)	0.0278 (10)	0.0270 (10)	−0.0080 (8)	−0.0019 (7)	−0.0091 (8)
C7	0.0263 (9)	0.0257 (10)	0.0257 (9)	−0.0093 (8)	−0.0008 (7)	−0.0079 (8)
C9	0.0336 (11)	0.0255 (11)	0.0439 (13)	−0.0040 (9)	−0.0051 (9)	−0.0034 (10)
C10	0.0412 (12)	0.0362 (12)	0.0365 (12)	−0.0103 (10)	−0.0107 (9)	−0.0148 (10)
C11	0.0260 (9)	0.0222 (9)	0.0245 (9)	−0.0022 (8)	−0.0055 (7)	−0.0055 (8)
C12	0.0350 (11)	0.0338 (11)	0.0275 (10)	−0.0081 (9)	−0.0046 (8)	−0.0132 (9)
C13	0.0373 (11)	0.0436 (13)	0.0238 (10)	−0.0105 (10)	−0.0023 (8)	−0.0096 (10)
C14	0.0348 (11)	0.0284 (11)	0.0324 (11)	−0.0098 (9)	−0.0056 (9)	−0.0012 (9)
C15	0.0381 (11)	0.0257 (10)	0.0368 (12)	−0.0077 (9)	−0.0075 (9)	−0.0111 (9)
C16	0.0339 (10)	0.0270 (10)	0.0274 (10)	−0.0043 (9)	−0.0038 (8)	−0.0111 (9)

Geometric parameters (Å, º)

I1—C4	2.101 (2)	C6—C9	1.502 (3)
I1—F1^{i}	3.4226 (16)	C8—C10	1.481 (3)
S1—O2	1.4874 (16)	C9—H9A	0.9800
S1—C1	1.754 (2)	C9—H9B	0.9800
S1—C11	1.797 (2)	C9—H9C	0.9800
F1—C14	1.360 (3)	C10—H10A	0.9800
O1—C8	1.374 (3)	C10—H10B	0.9800
O1—C7	1.377 (3)	C10—H10C	0.9800
C1—C8	1.349 (3)	C11—C16	1.378 (3)
C1—C2	1.450 (3)	C11—C12	1.388 (3)
C2—C7	1.394 (3)	C12—C13	1.381 (3)
C2—C3	1.395 (3)	C12—H12	0.9500
C3—C4	1.385 (3)	C13—C14	1.374 (4)
C3—H3	0.9500	C13—H13	0.9500
C4—C5	1.396 (3)	C14—C15	1.378 (3)
C5—C6	1.395 (3)	C15—C16	1.391 (3)
C5—H5	0.9500	C15—H15	0.9500

C6—C7	1.387 (3)	C16—H16	0.9500
C4—I1—F1^{i}	161.10 (6)	C6—C9—H9B	109.5
O2—S1—C1	107.49 (10)	H9A—C9—H9B	109.5
O2—S1—C11	106.55 (10)	C6—C9—H9C	109.5
C1—S1—C11	98.72 (9)	H9A—C9—H9C	109.5
C8—O1—C7	106.50 (16)	H9B—C9—H9C	109.5
C8—C1—C2	107.61 (18)	C8—C10—H10A	109.5
C8—C1—S1	123.72 (16)	C8—C10—H10B	109.5
C2—C1—S1	128.61 (16)	H10A—C10—H10B	109.5
C7—C2—C3	119.99 (19)	C8—C10—H10C	109.5
C7—C2—C1	104.33 (17)	H10A—C10—H10C	109.5
C3—C2—C1	135.67 (19)	H10B—C10—H10C	109.5
C4—C3—C2	116.1 (2)	C16—C11—C12	121.3 (2)
C4—C3—H3	122.0	C16—C11—S1	119.71 (16)
C2—C3—H3	122.0	C12—C11—S1	118.82 (17)
C3—C4—C5	123.2 (2)	C13—C12—C11	119.8 (2)
C3—C4—I1	119.05 (16)	C13—C12—H12	120.1
C5—C4—I1	117.79 (15)	C11—C12—H12	120.1
C6—C5—C4	121.5 (2)	C14—C13—C12	118.0 (2)
C6—C5—H5	119.3	C14—C13—H13	121.0
C4—C5—H5	119.3	C12—C13—H13	121.0
C7—C6—C5	114.6 (2)	F1—C14—C13	118.6 (2)
C7—C6—C9	122.6 (2)	F1—C14—C15	117.9 (2)
C5—C6—C9	122.8 (2)	C13—C14—C15	123.5 (2)
C1—C8—O1	110.86 (17)	C14—C15—C16	117.9 (2)
C1—C8—C10	133.1 (2)	C14—C15—H15	121.0
O1—C8—C10	116.02 (19)	C16—C15—H15	121.0
O1—C7—C6	124.59 (19)	C11—C16—C15	119.5 (2)
O1—C7—C2	110.69 (17)	C11—C16—H16	120.3
C6—C7—C2	124.7 (2)	C15—C16—H16	120.3
C6—C9—H9A	109.5		
O2—S1—C1—C8	139.60 (18)	C8—O1—C7—C2	−0.2 (2)
C11—S1—C1—C8	−109.88 (19)	C5—C6—C7—O1	−179.19 (19)
O2—S1—C1—C2	−37.4 (2)	C9—C6—C7—O1	1.3 (3)
C11—S1—C1—C2	73.1 (2)	C5—C6—C7—C2	1.0 (3)
C8—C1—C2—C7	0.5 (2)	C9—C6—C7—C2	−178.5 (2)
S1—C1—C2—C7	177.84 (16)	C3—C2—C7—O1	178.93 (17)
C8—C1—C2—C3	−178.4 (2)	C1—C2—C7—O1	−0.1 (2)
S1—C1—C2—C3	−1.0 (4)	C3—C2—C7—C6	−1.2 (3)
C7—C2—C3—C4	0.1 (3)	C1—C2—C7—C6	179.7 (2)
C1—C2—C3—C4	178.8 (2)	O2—S1—C11—C16	7.08 (19)
C2—C3—C4—C5	1.3 (3)	C1—S1—C11—C16	−104.19 (18)
C2—C3—C4—I1	−178.54 (14)	O2—S1—C11—C12	−168.41 (16)
C3—C4—C5—C6	−1.5 (3)	C1—S1—C11—C12	80.32 (18)
I1—C4—C5—C6	178.28 (16)	C16—C11—C12—C13	−0.2 (3)
C4—C5—C6—C7	0.4 (3)	S1—C11—C12—C13	175.24 (17)

C4—C5—C6—C9	179.8 (2)	C11—C12—C13—C14	−0.1 (3)
C2—C1—C8—O1	−0.6 (2)	C12—C13—C14—F1	−179.2 (2)
S1—C1—C8—O1	−178.17 (14)	C12—C13—C14—C15	1.3 (4)
C2—C1—C8—C10	−179.3 (2)	F1—C14—C15—C16	178.33 (19)
S1—C1—C8—C10	3.1 (4)	C13—C14—C15—C16	−2.2 (4)
C7—O1—C8—C1	0.5 (2)	C12—C11—C16—C15	−0.7 (3)
C7—O1—C8—C10	179.49 (18)	S1—C11—C16—C15	−176.06 (16)
C8—O1—C7—C6	179.94 (19)	C14—C15—C16—C11	1.8 (3)

Symmetry code: (i) −*x*+2, −*y*+2, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O2^{ii}	0.95	2.51	3.444 (3)	169
C9—H9*A*···O2^{iii}	0.98	2.48	3.437 (3)	165
C15—H15···O1^{iv}	0.95	2.49	3.342 (3)	149

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