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

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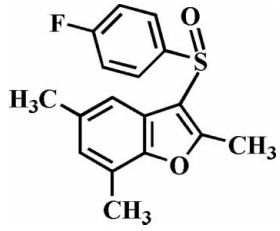
Key indicators: single-crystal X-ray study; T = 172 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.102; data-to-parameter ratio = 17.4.

In the title molecule, $C_{17}H_{15}FO_2S$, the O atom and the 4-fluorophenyl group of the 4-fluorophenylsulfinyl substituent lie on opposite sides of the benzofuran fragment. The mean planes of the benzofuran and 4-fluorophenyl fragments form a dihedral angle of 86.07 (4)°. In the crystal structure, weak intermolecular C—H···O hydrogen bonds link the molecules into centrosymmetric dimers, which are further linked *via* intermolecular C—H···π interactions.

Related literature

For the crystal structures of similar 2-methyl-3-phenylsulfinyl-1-benzofuran derivatives, see: Choi *et al.* (2007, 2008*a*,*b*). For the biological 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).



Experimental

Crystal data

$C_{17}H_{15}FO_2S$ M_r = 302.35
Triclinic, $P\overline{1}$
a = 6.2558 (2) Å
b = 11.1848 (3) Å
c = 11.9766 (5) Å
α = 110.355 (2)°
β = 99.448 (2)°
γ = 104.130 (1)°
V = 732.75 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 172 K
0.43 × 0.28 × 0.26 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.681, T_{max} = 0.746
12781 measured reflections
3350 independent reflections
3071 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.102
S = 1.03
3350 reflections
193 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

Cg is the centroid of the C2–C7 ring.

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13···O2^{i}	0.95	2.50	3.194 (2)	130
C11—H11B···Cg^{ii}	0.98	2.87	3.663 (2)	139

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

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: CV2691).

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supporting information

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

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S1. Comment

Molecules containing benzofuran skeleton show potent biological 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 2-methyl-3-phenylsulfinyl-1-benzofuran analogues (Choi *et al.*, 2007, 2008*a,b*), we report the crystal structure of the title compound (Fig. 1).

The benzofuran unit is essentially planar, with a mean deviation of 0.007 (1) Å from the least-squares plane defined by the nine constituent atoms. The 4-fluorophenyl ring is almost perpendicular to the plane of the benzofuran fragment [86.07 (4)°] and is tilted slightly towards it. The crystal packing (Fig. 2) is stabilized by a weak intermolecular C–H···O hydrogen bond between the 4-fluorophenyl H atom and the oxygen of the S═O unit (Table 1). The molecular packing (Fig. 2) is further stabilized by an intermolecular C–H···π interaction between the methyl H atom and the benzene ring of an adjacent benzofuran system, with a C11–H11B···Cg^{ii} (Table 1; Cg is the centroid of the C2-C7 benzene ring).

S2. Experimental

77% 3-Chloroperoxybenzoic acid (291 mg, 1.3 mmol) was added in small portions to a stirred solution of 3-(4-fluorophenylsulfanyl)-2,5,7-trimethyl-1-benzofuran (343 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 82%, m.p. 433-434 K; R_f = 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 ethyl acetate 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 1.5U_{eq}(C) for methyl H atoms.

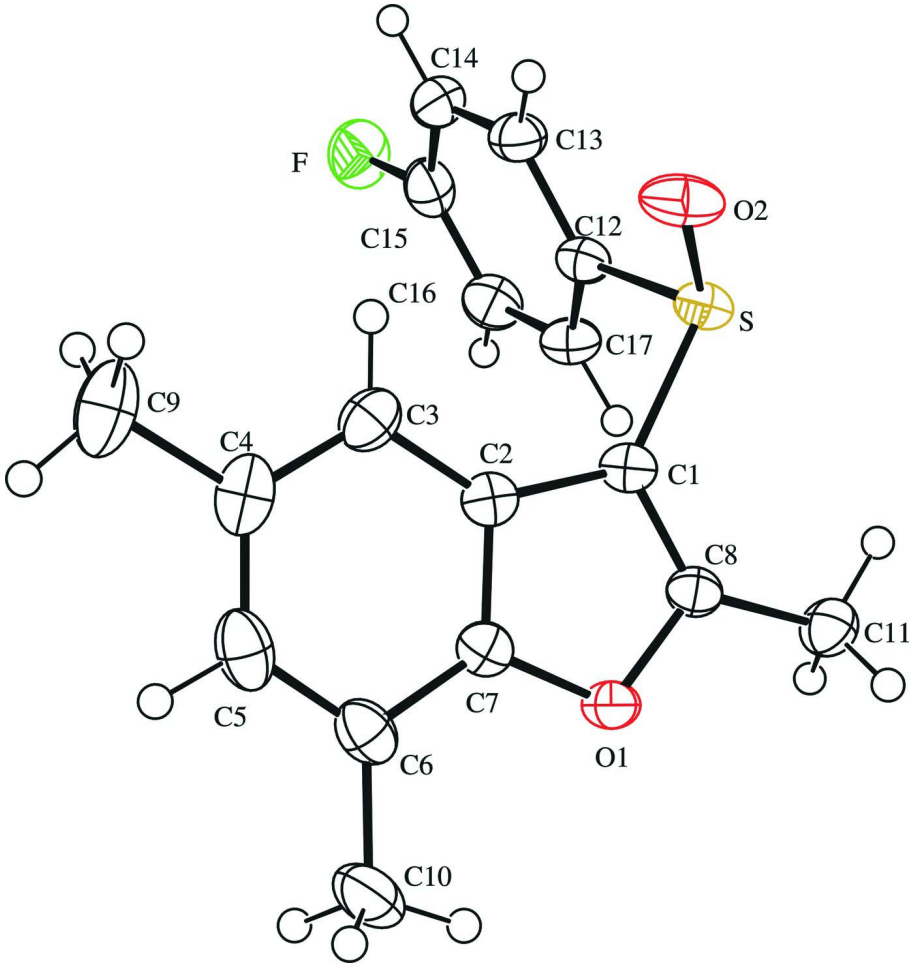

Figure 1

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.

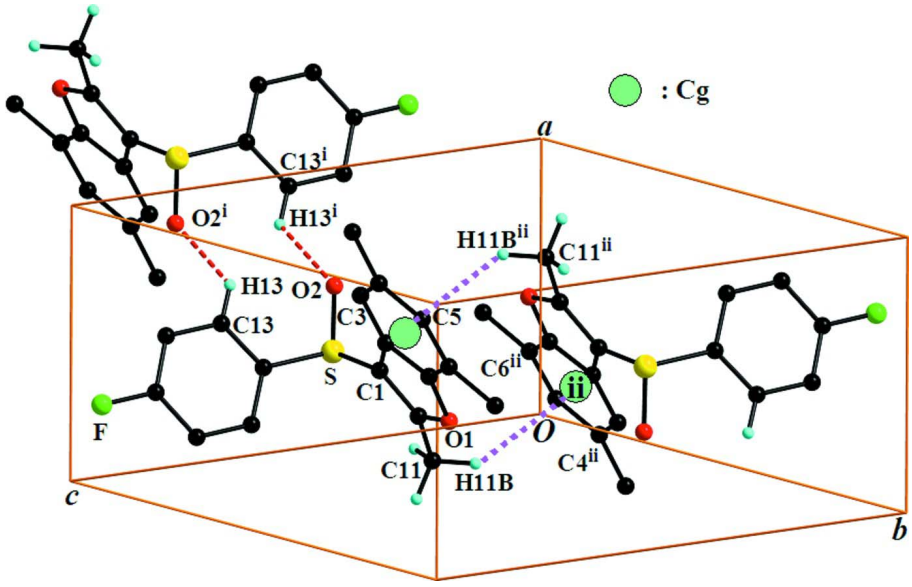

Figure 2

C–H···O and C–H···π interactions (dotted lines) in the crystal structure of the title compound. Cg denotes the ring (C2-C7) centroid. [Symmetry codes: (i) - x + 2, - y + 1, - z + 2; (ii) - x + 1, - y + 1, - z + 1.]

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

Crystal data

$C_{17}H_{15}FO_2S$
M_r = 302.35
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 6.2558 (2) Å
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c = 11.9766 (5) Å
α = 110.355 (2)°
β = 99.448 (2)°
γ = 104.130 (1)°
V = 732.75 (4) Å^3
Z = 2
F(000) = 316
D_x = 1.370 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9234 reflections
θ = 3.3–27.5°
μ = 0.23 mm^{-1}
T = 172 K
Block, colourless
0.43 × 0.28 × 0.26 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} = 0.681, T_{max} = 0.746
12781 measured reflections
3350 independent reflections
3071 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 27.5°, θ_{min} = 1.9°
h = −8→8
k = −14→14
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.102$
$S = 1.03$
3350 reflections
193 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
$w = 1/[\sigma^2(F_o^2) + (0.0539P)^2 + 0.3002P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S	0.61072 (6)	0.50406 (3)	0.83633 (3)	0.02837 (11)
F	0.28727 (19)	0.04833 (10)	0.97162 (10)	0.0498 (3)
O1	0.23445 (16)	0.38938 (10)	0.49810 (8)	0.0282 (2)
O2	0.86249 (18)	0.53145 (12)	0.85499 (10)	0.0409 (3)
C1	0.4760 (2)	0.42813 (13)	0.67695 (12)	0.0262 (3)
C2	0.5133 (2)	0.32114 (13)	0.58028 (12)	0.0265 (3)
C3	0.6563 (3)	0.24226 (15)	0.57323 (14)	0.0329 (3)
H3	0.7644	0.2546	0.6454	0.040*
C4	0.6370 (3)	0.14528 (15)	0.45835 (16)	0.0380 (3)
C5	0.4753 (3)	0.12862 (15)	0.35294 (15)	0.0391 (4)
H5	0.4639	0.0608	0.2755	0.047*
C6	0.3315 (3)	0.20587 (14)	0.35596 (13)	0.0337 (3)
C7	0.3589 (2)	0.30150 (13)	0.47270 (12)	0.0273 (3)
C8	0.3107 (2)	0.46565 (13)	0.62320 (11)	0.0261 (3)
C9	0.7900 (3)	0.05875 (18)	0.4476 (2)	0.0522 (5)
H9A	0.9355	0.1056	0.4379	0.078*
H9B	0.7145	−0.0271	0.3754	0.078*
H9C	0.8196	0.0416	0.5226	0.078*
C10	0.1578 (3)	0.19014 (17)	0.24463 (14)	0.0464 (4)
H10A	0.0033	0.1588	0.2532	0.070*
H10B	0.1734	0.1242	0.1698	0.070*
H10C	0.1834	0.2772	0.2382	0.070*
C11	0.1968 (3)	0.56646 (15)	0.67177 (13)	0.0337 (3)
H11A	0.0400	0.5202	0.6690	0.051*
H11B	0.1925	0.6186	0.6210	0.051*

H11C	0.2823	0.6274	0.7575	0.051*
C12	0.5089 (2)	0.35932 (13)	0.87080 (11)	0.0264 (3)
C13	0.6686 (2)	0.31841 (15)	0.92889 (12)	0.0310 (3)
H13	0.8279	0.3625	0.9454	0.037*
C14	0.5933 (3)	0.21171 (15)	0.96292 (13)	0.0350 (3)
H14	0.6994	0.1810	1.0023	0.042*
C15	0.3616 (3)	0.15211 (14)	0.93807 (13)	0.0348 (3)
C16	0.1997 (3)	0.19321 (15)	0.88255 (14)	0.0361 (3)
H16	0.0407	0.1499	0.8678	0.043*
C17	0.2753 (2)	0.29939 (15)	0.84890 (13)	0.0326 (3)
H17	0.1683	0.3309	0.8112	0.039*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.02613 (19)	0.03171 (19)	0.02281 (17)	0.00535 (14)	0.00233 (13)	0.01064 (13)
F	0.0627 (7)	0.0372 (5)	0.0530 (6)	0.0114 (5)	0.0166 (5)	0.0253 (4)
O1	0.0271 (5)	0.0333 (5)	0.0240 (4)	0.0108 (4)	0.0038 (4)	0.0120 (4)
O2	0.0236 (5)	0.0554 (7)	0.0374 (5)	0.0007 (5)	0.0002 (4)	0.0234 (5)
C1	0.0236 (6)	0.0306 (6)	0.0240 (6)	0.0080 (5)	0.0055 (5)	0.0117 (5)
C2	0.0237 (6)	0.0285 (6)	0.0278 (6)	0.0069 (5)	0.0080 (5)	0.0129 (5)
C3	0.0284 (7)	0.0352 (7)	0.0417 (8)	0.0131 (6)	0.0124 (6)	0.0200 (6)
C4	0.0403 (8)	0.0319 (7)	0.0522 (9)	0.0148 (6)	0.0261 (7)	0.0208 (7)
C5	0.0491 (9)	0.0294 (7)	0.0377 (7)	0.0090 (6)	0.0233 (7)	0.0098 (6)
C6	0.0389 (8)	0.0304 (7)	0.0275 (6)	0.0044 (6)	0.0116 (6)	0.0103 (5)
C7	0.0269 (7)	0.0285 (6)	0.0269 (6)	0.0073 (5)	0.0084 (5)	0.0122 (5)
C8	0.0246 (6)	0.0298 (6)	0.0241 (6)	0.0075 (5)	0.0063 (5)	0.0121 (5)
C9	0.0561 (11)	0.0433 (9)	0.0754 (13)	0.0285 (8)	0.0380 (10)	0.0278 (9)
C10	0.0601 (11)	0.0419 (8)	0.0245 (7)	0.0067 (8)	0.0047 (7)	0.0086 (6)
C11	0.0315 (7)	0.0368 (7)	0.0357 (7)	0.0160 (6)	0.0099 (6)	0.0145 (6)
C12	0.0257 (6)	0.0304 (6)	0.0210 (5)	0.0083 (5)	0.0049 (5)	0.0092 (5)
C13	0.0264 (7)	0.0374 (7)	0.0280 (6)	0.0119 (6)	0.0053 (5)	0.0118 (5)
C14	0.0393 (8)	0.0368 (7)	0.0314 (7)	0.0187 (6)	0.0065 (6)	0.0138 (6)
C15	0.0454 (9)	0.0280 (6)	0.0294 (6)	0.0100 (6)	0.0120 (6)	0.0105 (5)
C16	0.0296 (7)	0.0377 (7)	0.0359 (7)	0.0047 (6)	0.0078 (6)	0.0138 (6)
C17	0.0257 (7)	0.0396 (7)	0.0316 (7)	0.0100 (6)	0.0041 (5)	0.0154 (6)

Geometric parameters (Å, º)

S—O2	1.4919 (11)	C9—H9A	0.9800
S—C1	1.7521 (13)	C9—H9B	0.9800
S—C12	1.8013 (14)	C9—H9C	0.9800
F—C15	1.3591 (17)	C10—H10A	0.9800
O1—C8	1.3702 (15)	C10—H10B	0.9800
O1—C7	1.3814 (16)	C10—H10C	0.9800
C1—C8	1.3567 (19)	C11—H11A	0.9800
C1—C2	1.4450 (18)	C11—H11B	0.9800
C2—C7	1.3899 (19)	C11—H11C	0.9800

C2—C3	1.3942 (19)	C12—C13	1.3811 (19)
C3—C4	1.388 (2)	C12—C17	1.3885 (19)
C3—H3	0.9500	C13—C14	1.393 (2)
C4—C5	1.406 (2)	C13—H13	0.9500
C4—C9	1.509 (2)	C14—C15	1.372 (2)
C5—C6	1.387 (2)	C14—H14	0.9500
C5—H5	0.9500	C15—C16	1.378 (2)
C6—C7	1.3856 (19)	C16—C17	1.385 (2)
C6—C10	1.503 (2)	C16—H16	0.9500
C8—C11	1.4799 (19)	C17—H17	0.9500

O2—S—C1	108.53 (6)	H9A—C9—H9C	109.5
O2—S—C12	105.94 (6)	H9B—C9—H9C	109.5
C1—S—C12	97.96 (6)	C6—C10—H10A	109.5
C8—O1—C7	106.44 (10)	C6—C10—H10B	109.5
C8—C1—C2	107.66 (11)	H10A—C10—H10B	109.5
C8—C1—S	122.90 (10)	C6—C10—H10C	109.5
C2—C1—S	129.43 (10)	H10A—C10—H10C	109.5
C7—C2—C3	119.26 (13)	H10B—C10—H10C	109.5
C7—C2—C1	104.44 (11)	C8—C11—H11A	109.5
C3—C2—C1	136.30 (13)	C8—C11—H11B	109.5
C4—C3—C2	118.46 (14)	H11A—C11—H11B	109.5
C4—C3—H3	120.8	C8—C11—H11C	109.5
C2—C3—H3	120.8	H11A—C11—H11C	109.5
C3—C4—C5	119.76 (14)	H11B—C11—H11C	109.5
C3—C4—C9	119.71 (16)	C13—C12—C17	121.61 (13)
C5—C4—C9	120.53 (15)	C13—C12—S	118.26 (11)
C6—C5—C4	123.51 (14)	C17—C12—S	119.88 (11)
C6—C5—H5	118.2	C12—C13—C14	119.19 (13)
C4—C5—H5	118.2	C12—C13—H13	120.4
C7—C6—C5	114.30 (14)	C14—C13—H13	120.4
C7—C6—C10	121.21 (15)	C15—C14—C13	118.22 (14)
C5—C6—C10	124.49 (14)	C15—C14—H14	120.9
O1—C7—C6	124.51 (13)	C13—C14—H14	120.9
O1—C7—C2	110.78 (11)	F—C15—C14	118.43 (14)
C6—C7—C2	124.70 (13)	F—C15—C16	118.07 (14)
C1—C8—O1	110.67 (11)	C14—C15—C16	123.49 (14)
C1—C8—C11	133.59 (12)	C15—C16—C17	118.13 (14)
O1—C8—C11	115.72 (11)	C15—C16—H16	120.9
C4—C9—H9A	109.5	C17—C16—H16	120.9
C4—C9—H9B	109.5	C16—C17—C12	119.34 (14)
H9A—C9—H9B	109.5	C16—C17—H17	120.3
C4—C9—H9C	109.5	C12—C17—H17	120.3

O2—S—C1—C8	−134.93 (12)	C1—C2—C7—O1	0.50 (14)
C12—S—C1—C8	115.24 (12)	C3—C2—C7—C6	1.2 (2)
O2—S—C1—C2	44.44 (14)	C1—C2—C7—C6	−178.86 (13)
C12—S—C1—C2	−65.40 (13)	C2—C1—C8—O1	0.87 (15)

C8—C1—C2—C7	−0.82 (15)	S—C1—C8—O1	−179.65 (9)
S—C1—C2—C7	179.74 (11)	C2—C1—C8—C11	179.02 (14)
C8—C1—C2—C3	179.13 (15)	S—C1—C8—C11	−1.5 (2)
S—C1—C2—C3	−0.3 (2)	C7—O1—C8—C1	−0.55 (15)
C7—C2—C3—C4	−0.7 (2)	C7—O1—C8—C11	−179.07 (11)
C1—C2—C3—C4	179.31 (15)	O2—S—C12—C13	12.66 (12)
C2—C3—C4—C5	−0.2 (2)	C1—S—C12—C13	124.61 (11)
C2—C3—C4—C9	179.74 (13)	O2—S—C12—C17	−172.98 (11)
C3—C4—C5—C6	0.8 (2)	C1—S—C12—C17	−61.03 (12)
C9—C4—C5—C6	−179.12 (14)	C17—C12—C13—C14	2.0 (2)
C4—C5—C6—C7	−0.4 (2)	S—C12—C13—C14	176.26 (10)
C4—C5—C6—C10	179.89 (15)	C12—C13—C14—C15	−0.6 (2)
C8—O1—C7—C6	179.37 (13)	C13—C14—C15—F	−179.71 (12)
C8—O1—C7—C2	0.00 (14)	C13—C14—C15—C16	−0.8 (2)
C5—C6—C7—O1	−179.85 (12)	F—C15—C16—C17	179.68 (13)
C10—C6—C7—O1	−0.2 (2)	C14—C15—C16—C17	0.7 (2)
C5—C6—C7—C2	−0.6 (2)	C15—C16—C17—C12	0.7 (2)
C10—C6—C7—C2	179.13 (14)	C13—C12—C17—C16	−2.0 (2)
C3—C2—C7—O1	−179.46 (12)	S—C12—C17—C16	−176.20 (11)

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C2–C7 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13···O2^{i}	0.95	2.50	3.194 (2)	130
C11—H11*B*···*Cg*^{ii}	0.98	2.87	3.663 (2)	139

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