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3-(4-Fluorophenylsulfinyl)-2,4,5,6-tetramethyl-1-benzofuran

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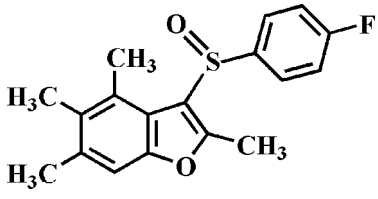
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.094; data-to-parameter ratio = 18.5.

In the title compound, $C_{18}H_{17}FO_2S$, the dihedral angle between the plane of the benzofuran ring system (r.m.s. deviation = 0.013 Å) and that of the 4-fluorophenyl ring is 74.30 (5)°. In the crystal, weak C—H···O and C—H···F hydrogen bonds link the molecules into columns extending in direction [100].

Related literature

For the crystal structures of related compounds, see: Choi *et al.* (2012); Seo *et al.* (2011*a*,*b*).



Experimental

Crystal data

$C_{18}H_{17}FO_2S$
M_r = 316.38
Orthorhombic, $Pna2_1$
a = 7.8869 (2) Å
b = 11.0042 (2) Å
c = 17.5181 (4) Å
V = 1520.38 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 173 K
0.67 × 0.32 × 0.27 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.862, T_{max} = 0.941
13898 measured reflections
3761 independent reflections
3369 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.094
S = 1.05
3761 reflections
203 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Absolute structure: Flack (1983), 1812 Friedel pairs
Absolute structure parameter: −0.02 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
C15—H15···O2^{i}	0.95	2.31	3.247 (3)	169
C17—H17···F1^{ii}	0.95	2.50	3.083 (2)	120

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

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 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: CV5468).

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supporting information

Acta Cryst. (2014). E**70**, o830 [https://doi.org/10.1107/S1600536814014822]

3-(4-Fluorophenylsulfinyl)-2,4,5,6-tetramethyl-1-benzofuran

Hong Dae Choi, Pil Ja Seo and Uk Lee

S1. Comment

As a part of our ongoing project of 2,4,5,6-tetramethyl-1-benzofuran derivatives containing phenylsulfinyl (Seo *et al.*, 2011*a*), 3-fluorophenylsulfinyl (Seo *et al.*, 2011*b*) and 2-fluorophenylsulfinyl (Choi *et al.*, 2012) substituents in 3-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 0.013 (2) Å from the least-squares plane defined by the nine constituent atoms. The 4-fluorophenyl ring is essentially planar, with a mean deviation of 0.002 (1) Å from the least-squares plane defined by the six constituent atoms. The dihedral angle formed by the benzofuran ring system and the 4-fluorophenyl ring is 74.30 (5)°. In the crystal structure (Fig. 2), weak intermolecular C—H···O and C—H···F hydrogen bonds (Table 1) link the molecules into columns extended in [100].

S2. Experimental

3-Chloroperoxybenzoic acid (77%, 269 mg, 1.2 mmol) was added in small portions to a stirred solution of 3-(4-fluorophenylsulfanyl)-2,4,5,6-tetramethyl-1-benzofuran (330 mg, 1.1 mmol) in dichloromethane (35 mL) at 273 K. After being stirred at room temperature for 5h, 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, 2:1 *v*/*v*) to afford the title compound as a colorless solid [yield 74%, m.p. 443–444 K; R_f = 0.59 (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 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, 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's command AFIX 137 (Sheldrick, 2008).

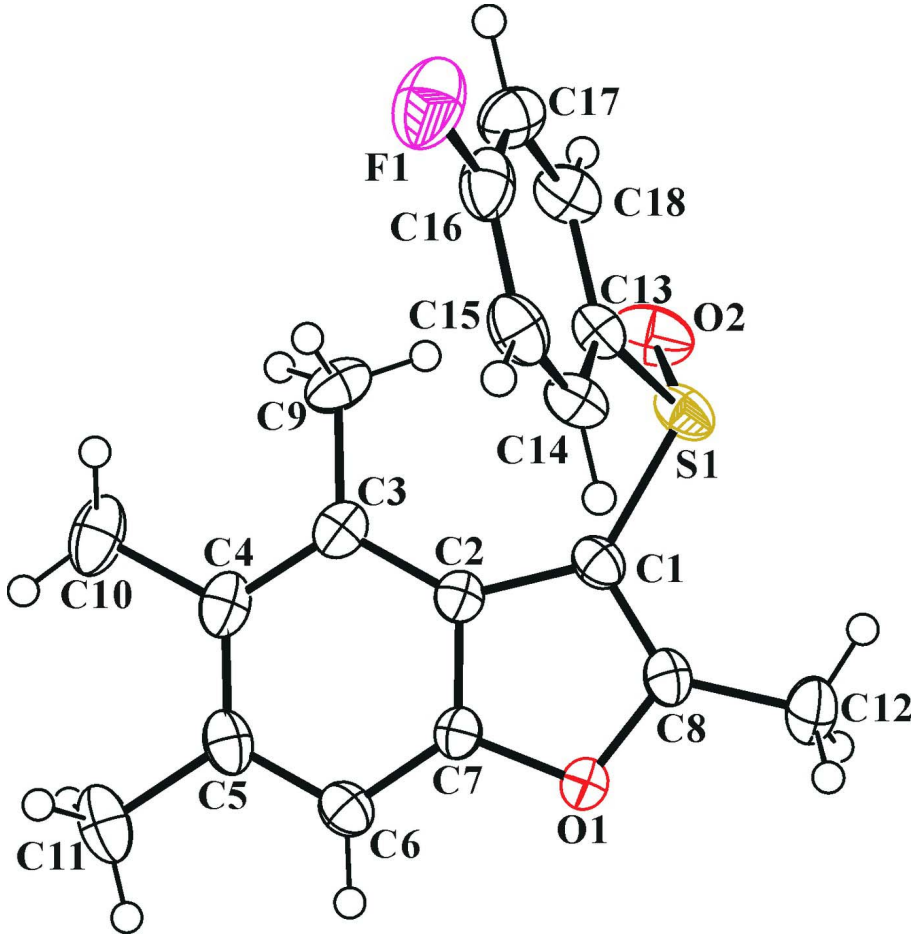

Figure 1

The molecular structure of the title compound 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.

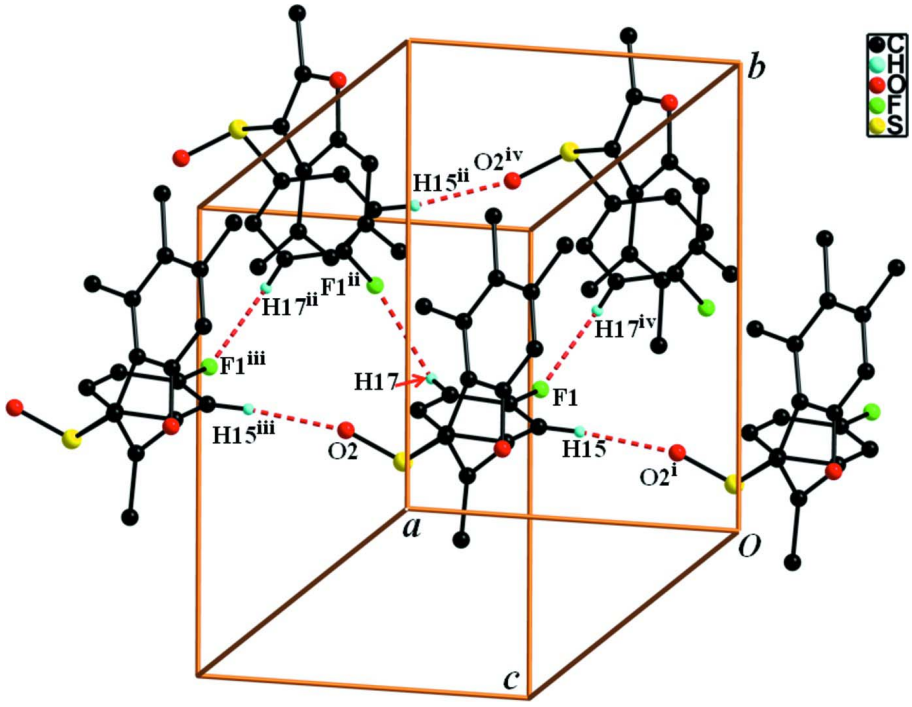

Figure 2

A portion of the crystal packing showing the C—H···O and C—H···F interactions as dotted lines. H atoms non-participating in hydrogen-bonding were omitted for clarity. [Symmetry codes: (i) *x* - 1, *y*, *z*; (ii) *x* + 1/2, - *y* + 3/2, *z*; (iii) *x* + 1, *y*, *z*; (iv) *x* - 1/2, - *y* + 3/2, *z*.]

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Crystal data

$C_{18}H_{17}FO_2S$
$M_r = 316.38$
Orthorhombic, $Pna2_1$
Hall symbol: P 2c -2n
$a = 7.8869$ (2) Å
$b = 11.0042$ (2) Å
$c = 17.5181$ (4) Å
$V = 1520.38$ (6) Å^3
$Z = 4$
$F(000) = 664$
$D_x = 1.382$ Mg m^{-3}
Melting point = 444–443 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3560 reflections
$\theta = 2.2$–$27.1°$
$\mu = 0.23$ mm^{-1}
$T = 173$ K
Block, colourless
0.67 × 0.32 × 0.27 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)
$T_{min} = 0.862$, $T_{max} = 0.941$
13898 measured reflections
3761 independent reflections
3369 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.2°$
$h = -10 \rightarrow 10$
$k = -13 \rightarrow 14$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.094$
$S = 1.05$
3761 reflections
203 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0506P)^2 + 0.0839P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}
Absolute structure: Flack (1983), 1812 Friedel pairs
Absolute structure parameter: −0.02 (7)

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}
S1	0.51230 (6)	0.40717 (5)	0.79515 (3)	0.03465 (13)
F1	−0.01469 (18)	0.65099 (13)	0.97462 (9)	0.0533 (4)
O1	0.34363 (18)	0.36849 (11)	0.58751 (7)	0.0285 (3)
O2	0.67580 (19)	0.47156 (18)	0.80968 (9)	0.0501 (4)
C1	0.4429 (3)	0.43098 (17)	0.70108 (10)	0.0284 (4)
C2	0.4022 (2)	0.53931 (16)	0.65691 (10)	0.0258 (4)
C3	0.4111 (2)	0.66591 (17)	0.66725 (10)	0.0290 (4)
C4	0.3498 (3)	0.73996 (17)	0.60784 (11)	0.0322 (4)
C5	0.2879 (3)	0.68941 (18)	0.53945 (11)	0.0320 (4)
C6	0.2837 (3)	0.56431 (17)	0.52893 (11)	0.0303 (4)
H6	0.2432	0.5291	0.4829	0.036*
C7	0.3410 (2)	0.49393 (15)	0.58838 (10)	0.0248 (4)
C8	0.4051 (2)	0.33292 (16)	0.65686 (10)	0.0292 (4)
C9	0.4869 (3)	0.7198 (2)	0.73820 (12)	0.0379 (5)
H9A	0.5548	0.6581	0.7645	0.057*
H9B	0.3960	0.7478	0.7720	0.057*
H9C	0.5595	0.7887	0.7244	0.057*
C10	0.3533 (3)	0.87599 (18)	0.61812 (15)	0.0465 (6)
H10A	0.4666	0.9068	0.6057	0.070*
H10B	0.3260	0.8961	0.6712	0.070*
H10C	0.2696	0.9135	0.5842	0.070*
C11	0.2277 (3)	0.7687 (2)	0.47404 (14)	0.0458 (6)
H11A	0.1927	0.7172	0.4312	0.069*

H11B	0.3202	0.8222	0.4577	0.069*
H11C	0.1313	0.8181	0.4909	0.069*
C12	0.4159 (3)	0.19987 (17)	0.66832 (12)	0.0401 (5)
H12A	0.5087	0.1670	0.6374	0.060*
H12B	0.3088	0.1620	0.6528	0.060*
H12C	0.4373	0.1825	0.7223	0.060*
C13	0.3505 (2)	0.49187 (17)	0.84426 (10)	0.0285 (4)
C14	0.1820 (3)	0.47592 (19)	0.82583 (10)	0.0318 (4)
H14	0.1518	0.4272	0.7832	0.038*
C15	0.0572 (3)	0.53021 (19)	0.86895 (11)	0.0352 (4)
H15	−0.0593	0.5205	0.8567	0.042*
C16	0.1075 (3)	0.59918 (18)	0.93053 (12)	0.0360 (5)
C17	0.2734 (3)	0.61688 (18)	0.94997 (13)	0.0381 (5)
H17	0.3029	0.6655	0.9928	0.046*
C18	0.3969 (3)	0.56253 (19)	0.90609 (11)	0.0349 (5)
H18	0.5132	0.5735	0.9182	0.042*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0286 (2)	0.0469 (3)	0.02846 (19)	0.0087 (2)	−0.0041 (2)	0.0047 (2)
F1	0.0522 (9)	0.0416 (8)	0.0662 (9)	0.0090 (6)	0.0207 (7)	−0.0019 (7)
O1	0.0321 (7)	0.0232 (6)	0.0300 (6)	0.0017 (6)	−0.0017 (6)	−0.0008 (5)
O2	0.0249 (7)	0.0827 (12)	0.0429 (9)	0.0025 (8)	−0.0057 (6)	−0.0053 (8)
C1	0.0249 (9)	0.0339 (10)	0.0264 (8)	0.0038 (8)	−0.0003 (7)	0.0030 (7)
C2	0.0251 (10)	0.0258 (8)	0.0265 (8)	−0.0008 (7)	0.0039 (7)	0.0012 (7)
C3	0.0256 (10)	0.0282 (9)	0.0332 (9)	−0.0029 (8)	0.0096 (8)	−0.0016 (7)
C4	0.0300 (11)	0.0260 (9)	0.0406 (10)	−0.0015 (8)	0.0131 (9)	0.0041 (7)
C5	0.0287 (10)	0.0330 (10)	0.0344 (9)	0.0043 (8)	0.0077 (8)	0.0104 (8)
C6	0.0285 (10)	0.0353 (10)	0.0271 (8)	0.0013 (8)	0.0019 (8)	0.0032 (8)
C7	0.0230 (9)	0.0237 (8)	0.0277 (8)	0.0013 (7)	0.0031 (7)	0.0003 (7)
C8	0.0284 (10)	0.0274 (9)	0.0318 (9)	0.0047 (8)	0.0006 (8)	0.0040 (7)
C9	0.0397 (12)	0.0354 (11)	0.0385 (10)	−0.0108 (9)	0.0074 (9)	−0.0103 (9)
C10	0.0478 (14)	0.0286 (10)	0.0631 (14)	−0.0003 (10)	0.0213 (12)	0.0057 (10)
C11	0.0433 (13)	0.0459 (12)	0.0481 (12)	0.0081 (11)	0.0053 (10)	0.0209 (10)
C12	0.0431 (13)	0.0280 (10)	0.0491 (11)	0.0056 (9)	−0.0008 (10)	0.0028 (9)
C13	0.0283 (10)	0.0322 (9)	0.0249 (7)	0.0017 (8)	−0.0009 (8)	0.0055 (7)
C14	0.0288 (10)	0.0411 (11)	0.0255 (8)	−0.0032 (9)	−0.0038 (7)	0.0039 (7)
C15	0.0258 (10)	0.0417 (11)	0.0382 (10)	0.0012 (9)	−0.0017 (8)	0.0119 (9)
C16	0.0404 (13)	0.0286 (9)	0.0390 (10)	0.0021 (9)	0.0114 (9)	0.0078 (8)
C17	0.0432 (13)	0.0345 (10)	0.0366 (9)	−0.0108 (9)	0.0053 (10)	−0.0032 (8)
C18	0.0267 (10)	0.0456 (12)	0.0323 (9)	−0.0081 (9)	−0.0024 (8)	0.0038 (8)

Geometric parameters (Å, º)

S1—O2	1.4932 (17)	C9—H9C	0.9800
S1—C1	1.7560 (18)	C10—H10A	0.9800
S1—C13	1.799 (2)	C10—H10B	0.9800

F1—C16	1.361 (3)	C10—H10C	0.9800
O1—C8	1.365 (2)	C11—H11A	0.9800
O1—C7	1.381 (2)	C11—H11B	0.9800
C1—C8	1.361 (3)	C11—H11C	0.9800
C1—C2	1.457 (3)	C12—H12A	0.9800
C2—C7	1.387 (2)	C12—H12B	0.9800
C2—C3	1.407 (3)	C12—H12C	0.9800
C3—C4	1.408 (3)	C13—C14	1.379 (3)
C3—C9	1.501 (3)	C13—C18	1.383 (3)
C4—C5	1.408 (3)	C14—C15	1.377 (3)
C4—C10	1.508 (3)	C14—H14	0.9500
C5—C6	1.389 (3)	C15—C16	1.377 (3)
C5—C11	1.517 (3)	C15—H15	0.9500
C6—C7	1.374 (2)	C16—C17	1.366 (3)
C6—H6	0.9500	C17—C18	1.377 (3)
C8—C12	1.480 (3)	C17—H17	0.9500
C9—H9A	0.9800	C18—H18	0.9500
C9—H9B	0.9800		

O2—S1—C1	110.99 (10)	C4—C10—H10B	109.5
O2—S1—C13	106.57 (10)	H10A—C10—H10B	109.5
C1—S1—C13	98.65 (9)	C4—C10—H10C	109.5
C8—O1—C7	106.39 (13)	H10A—C10—H10C	109.5
C8—C1—C2	107.33 (16)	H10B—C10—H10C	109.5
C8—C1—S1	118.95 (14)	C5—C11—H11A	109.5
C2—C1—S1	133.56 (14)	C5—C11—H11B	109.5
C7—C2—C3	119.05 (16)	H11A—C11—H11B	109.5
C7—C2—C1	104.00 (15)	C5—C11—H11C	109.5
C3—C2—C1	136.95 (17)	H11A—C11—H11C	109.5
C2—C3—C4	117.44 (17)	H11B—C11—H11C	109.5
C2—C3—C9	121.18 (18)	C8—C12—H12A	109.5
C4—C3—C9	121.36 (17)	C8—C12—H12B	109.5
C3—C4—C5	121.30 (16)	H12A—C12—H12B	109.5
C3—C4—C10	118.68 (19)	C8—C12—H12C	109.5
C5—C4—C10	120.02 (19)	H12A—C12—H12C	109.5
C6—C5—C4	120.84 (17)	H12B—C12—H12C	109.5
C6—C5—C11	117.55 (19)	C14—C13—C18	120.66 (19)
C4—C5—C11	121.59 (18)	C14—C13—S1	120.38 (15)
C7—C6—C5	116.75 (18)	C18—C13—S1	118.57 (15)
C7—C6—H6	121.6	C15—C14—C13	120.33 (18)
C5—C6—H6	121.6	C15—C14—H14	119.8
C6—C7—O1	124.06 (16)	C13—C14—H14	119.8
C6—C7—C2	124.57 (16)	C14—C15—C16	117.6 (2)
O1—C7—C2	111.37 (14)	C14—C15—H15	121.2
C1—C8—O1	110.92 (15)	C16—C15—H15	121.2
C1—C8—C12	133.95 (18)	F1—C16—C17	118.5 (2)
O1—C8—C12	115.13 (16)	F1—C16—C15	118.1 (2)
C3—C9—H9A	109.5	C17—C16—C15	123.4 (2)

C3—C9—H9B	109.5	C16—C17—C18	118.4 (2)
H9A—C9—H9B	109.5	C16—C17—H17	120.8
C3—C9—H9C	109.5	C18—C17—H17	120.8
H9A—C9—H9C	109.5	C17—C18—C13	119.6 (2)
H9B—C9—H9C	109.5	C17—C18—H18	120.2
C4—C10—H10A	109.5	C13—C18—H18	120.2

O2—S1—C1—C8	126.77 (17)	C8—O1—C7—C2	0.87 (19)
C13—S1—C1—C8	−121.68 (17)	C3—C2—C7—C6	−1.4 (3)
O2—S1—C1—C2	−58.4 (2)	C1—C2—C7—C6	178.85 (18)
C13—S1—C1—C2	53.1 (2)	C3—C2—C7—O1	178.97 (16)
C8—C1—C2—C7	0.3 (2)	C1—C2—C7—O1	−0.7 (2)
S1—C1—C2—C7	−174.90 (17)	C2—C1—C8—O1	0.2 (2)
C8—C1—C2—C3	−179.3 (2)	S1—C1—C8—O1	176.24 (13)
S1—C1—C2—C3	5.5 (4)	C2—C1—C8—C12	−179.5 (2)
C7—C2—C3—C4	2.7 (3)	S1—C1—C8—C12	−3.5 (3)
C1—C2—C3—C4	−177.7 (2)	C7—O1—C8—C1	−0.6 (2)
C7—C2—C3—C9	−175.95 (18)	C7—O1—C8—C12	179.11 (17)
C1—C2—C3—C9	3.6 (3)	O2—S1—C13—C14	162.46 (14)
C2—C3—C4—C5	−2.4 (3)	C1—S1—C13—C14	47.42 (16)
C9—C3—C4—C5	176.21 (18)	O2—S1—C13—C18	−24.61 (18)
C2—C3—C4—C10	178.38 (18)	C1—S1—C13—C18	−139.66 (16)
C9—C3—C4—C10	−3.0 (3)	C18—C13—C14—C15	−0.1 (3)
C3—C4—C5—C6	0.8 (3)	S1—C13—C14—C15	172.70 (15)
C10—C4—C5—C6	179.98 (19)	C13—C14—C15—C16	−0.5 (3)
C3—C4—C5—C11	−177.67 (19)	C14—C15—C16—F1	−178.51 (17)
C10—C4—C5—C11	1.5 (3)	C14—C15—C16—C17	0.7 (3)
C4—C5—C6—C7	0.6 (3)	F1—C16—C17—C18	178.83 (18)
C11—C5—C6—C7	179.10 (18)	C15—C16—C17—C18	−0.4 (3)
C5—C6—C7—O1	179.29 (17)	C16—C17—C18—C13	−0.2 (3)
C5—C6—C7—C2	−0.2 (3)	C14—C13—C18—C17	0.4 (3)
C8—O1—C7—C6	−178.71 (18)	S1—C13—C18—C17	−172.50 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15—H15···O2^{i}	0.95	2.31	3.247 (3)	169
C17—H17···F1^{ii}	0.95	2.50	3.083 (2)	120

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