

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

5-Fluoro-3-(3-fluorophenylsulfonyl)-2-methyl-1-benzofuran

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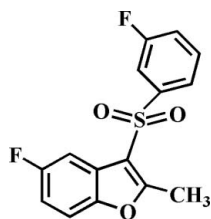
Received 9 July 2012; accepted 13 July 2012

Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; disorder in main residue; R factor = 0.037; wR factor = 0.104; data-to-parameter ratio = 16.2.

In the title compound, $\text{C}_{15}\text{H}_{10}\text{F}_2\text{O}_3\text{S}$, the 3-fluorophenyl ring makes a dihedral angle of $79.51(6)^\circ$ with the mean plane [r.m.s. deviation = $0.006(2)$ Å] of the benzofuran fragment. In the crystal, molecules are linked by weak $\text{C}-\text{H}\cdots\text{F}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds. The crystal structure also exhibits slipped $\pi-\pi$ interactions between the benzene and furan rings of neighbouring molecules [centroid-centroid distances = $3.563(3)$ and $3.820(3)$ Å and slippages of $0.358(3)$ and $1.551(3)$ Å]. In the 3-fluorophenyl ring, the F atom is disordered over two positions with site-occupancy factors of $0.887(3)$ and $0.113(3)$.

Related literature

For background information and the crystal structures of related compounds, see: Choi *et al.* (2010, 2012).



Experimental

Crystal data

 $\text{C}_{15}\text{H}_{10}\text{F}_2\text{O}_3\text{S}$
 $M_r = 308.29$

Monoclinic, $P2_1/n$
 $a = 7.2772(2)$ Å
 $b = 11.0972(3)$ Å
 $c = 16.5625(5)$ Å
 $\beta = 100.698(2)^\circ$
 $V = 1314.28(6)$ Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.28$ mm⁻¹
 $T = 296$ K
 $0.30 \times 0.22 \times 0.20$ mm

Data collection

Bruker SMART APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2009)
 $T_{\min} = 0.663$, $T_{\max} = 0.746$

12792 measured reflections
 3275 independent reflections
 2732 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.104$
 $S = 1.04$
 3275 reflections
 202 parameters

14 restraints
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.36$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.31$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C5}-\text{H5}\cdots\text{F2}^i$	0.93	2.45	3.302(2)	153
$\text{C15}-\text{H15}\cdots\text{O3}^{ii}$	0.93	2.47	3.3754(19)	165

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

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 (Farrugia, 1997) and DIAMOND (Brandenburg, 1998); software used to prepare material for publication: SHELXL97.

This work was supported by the Blue-Bio Industry Regional Innovation Center (RIC08-06-07) at Donggeui University as an RIC program under the Ministry of Knowledge Economy and Busan city.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2432).

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supporting information

Acta Cryst. (2012). E68, o2477 [https://doi.org/10.1107/S1600536812031972]

5-Fluoro-3-(3-fluorophenylsulfonyl)-2-methyl-1-benzofuran**Hong Dae Choi, Pil Ja Seo and Uk Lee****S1. Comment**

As a part of our continuing study of 5-fluoro-2-methyl-1-benzofuran derivatives containing 3-(4-fluorophenylsulfonyl) (Choi *et al.*, 2010) and 3-(4-methylphenylsulfonyl) (Choi *et al.*, 2012) substituents, we report herein the crystal structure of the title compound.

In the title molecule (Fig. 1), the benzofuran unit is essentially planar, with a mean deviation of 0.006 (2) Å from the least-squares plane defined by the nine constituent atoms. In the 3-fluorophenyl ring, the F atom is disordered over two positions with site-occupancy factors, from refinement of 0.887 (3) (part A) and 0.113 (3) (part B). The dihedral angle between the 3-fluorophenyl ring and the mean plane of the benzofuran ring is 79.51 (6)°. In the crystal structure (Fig. 2), molecules are connected by weak intermolecular C–H···F and C–H···O hydrogen bonds (Table 1). The crystal packing (Fig. 3) also exhibits slipped π – π interactions between the benzene and furan rings of neighbouring molecules, with Cg1···Cg2ⁱⁱⁱ and Cg1···Cg2^{iv} distances of 3.563 (3) Å & 3.820 (3) Å, and interplanar distances of 3.545 (3) Å & 3.491 (3) Å resulting in slippages of 0.358 (3) Å & 1.551 (3) Å (Cg1 and Cg2 are the centroids of the C2–C7 benzene ring and the C1/C2/C7/O1/C8 furan ring, respectively).

S2. Experimental

3-Chloroperoxybenzoic acid (77%, 515 mg, 2.3 mmol) was added in small portions to a stirred solution of 5-fluoro-3-(3-fluorophenylsulfonyl)-2-methyl-1-benzofuran (304 mg, 1.1 mmol) in dichloromethane (50 mL) at 273 K. After being stirred at room temperature for 10h, 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 (benzene) to afford the title compound as a colorless solid [yield 73%, m.p. 443–444 K; R_f = 0.66 (benzene)]. 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 = 0.93 Å for aryl and 0.96 Å for methyl H atoms. $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aryl and $1.5U_{\text{eq}}(\text{C})$ for methyl H atoms. The positions of methyl hydrogens were optimized rotationally. The F2 atom of the 3-fluorophenyl ring is disordered over two positions with site occupancy factors, from refinement of 0.887 (3) (part A) and 0.113 (3) (part B). The distance of equivalent C–F pairs were restrained to 1.330 (5) Å using the SHELXL-97 command DFIX, and displacement ellipsoids of F2 set were restrained using the SHELXL-97 command ISOR.

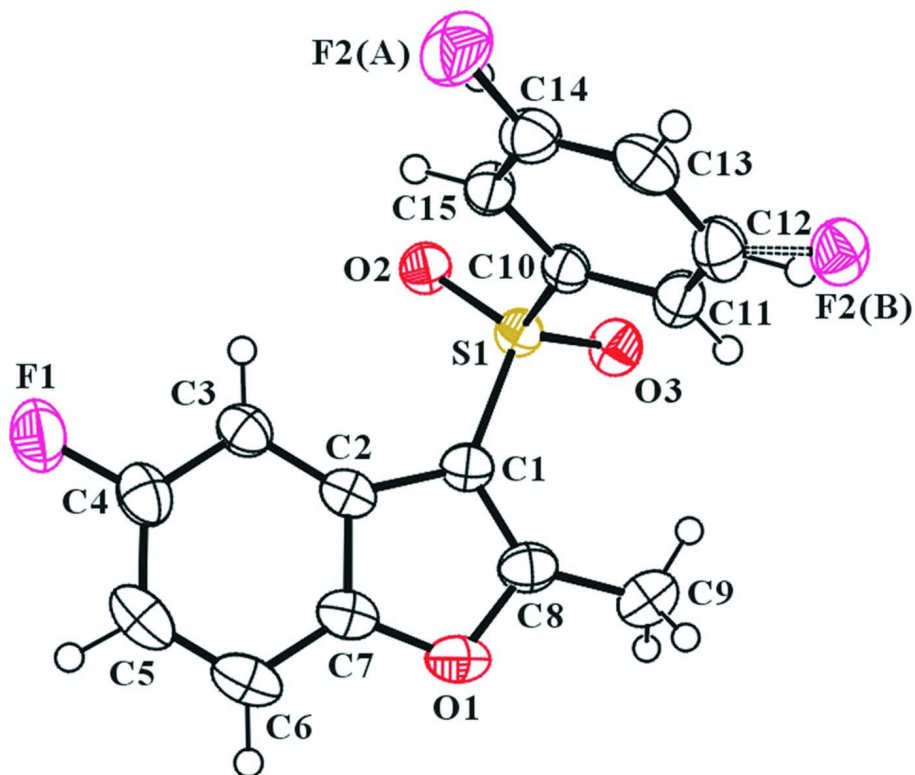


Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are presented as small spheres of arbitrary radius. The F atom of the 3-fluorophenyl ring is disordered over two positions with refined site occupancy factors, of 0.887 (3) (part A) and 0.113 (3) (part B).

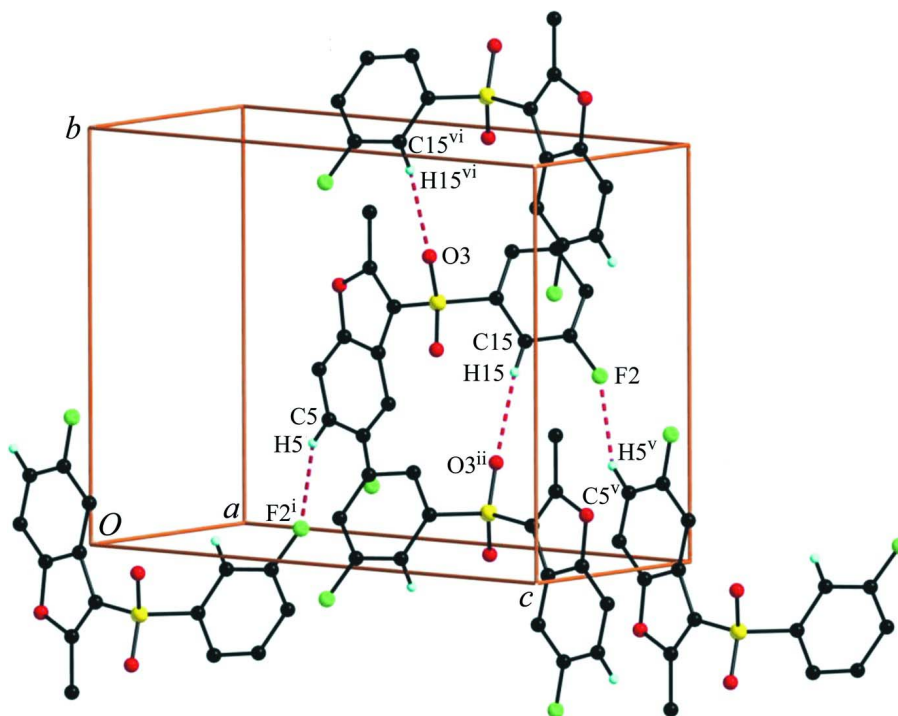


Figure 2

A view of the C–H···F and C–H···O interactions (dotted lines) in the crystal structure of the title compound. H atoms not participating in hydrogen-bonding were omitted for clarity. [Symmetry codes: (i) $x - 1/2, -y + 1/2, z - 1/2$; (ii) $-x + 1/2, y - 1/2, -z + 3/2$; (v) $x + 1/2, -y + 1/2, z + 1/2$; (vi) $-x + 1/2, y + 1/2, -z + 3/2$.]

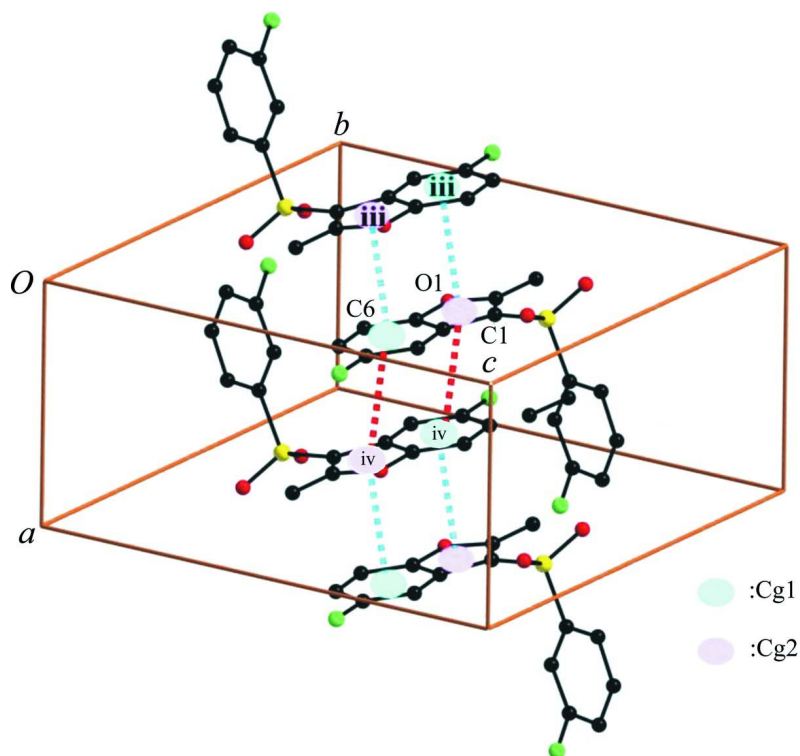


Figure 3

A view of the π - π interactions (dotted lines) in the crystal structure of the title compound. All H atoms were omitted for clarity. [Symmetry codes: (iii) $-x, -y + 1, -z + 1$; (iv) $-x + 1, -y + 1, -z + 1$]

5-Fluoro-3-(3-fluorophenylsulfonyl)-2-methyl-1-benzofuran

Crystal data

$C_{15}H_{10}F_2O_3S$

$M_r = 308.29$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1n$

$a = 7.2772\ (2)\ \text{\AA}$

$b = 11.0972\ (3)\ \text{\AA}$

$c = 16.5625\ (5)\ \text{\AA}$

$\beta = 100.698\ (2)^\circ$

$V = 1314.28\ (6)\ \text{\AA}^3$

$Z = 4$

$F(000) = 632$

$D_x = 1.558\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 3933 reflections

$\theta = 2.2\text{--}28.2^\circ$

$\mu = 0.28\ \text{mm}^{-1}$

$T = 296\ \text{K}$

Block, colourless

$0.30 \times 0.22 \times 0.20\ \text{mm}$

Data collection

Bruker SMART APEXII CCD
diffractometer

Radiation source: rotating anode

Graphite multilayer monochromator

Detector resolution: $10.0\ \text{pixels mm}^{-1}$

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2009)

$T_{\min} = 0.663, T_{\max} = 0.746$

12792 measured reflections

3275 independent reflections

2732 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.032$

$\theta_{\max} = 28.3^\circ, \theta_{\min} = 2.2^\circ$

$h = -9 \rightarrow 9$

$k = -14 \rightarrow 14$

$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.104$
 $S = 1.04$
 3275 reflections
 202 parameters
 14 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
 $w = 1/[\sigma^2(F_o^2) + (0.0548P)^2 + 0.3108P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.36 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.31 \text{ e } \text{\AA}^{-3}$
 Extinction correction: *SHELXL97* (Sheldrick,
 2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0088 (14)

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
S1	0.21640 (5)	0.63440 (3)	0.70540 (2)	0.03087 (13)	
F1	0.25059 (17)	0.17801 (10)	0.54399 (7)	0.0617 (3)	
O1	0.25258 (16)	0.65312 (11)	0.47256 (7)	0.0437 (3)	
O2	0.13169 (15)	0.52825 (10)	0.73181 (7)	0.0373 (3)	
O3	0.13613 (15)	0.75027 (10)	0.71521 (7)	0.0407 (3)	
C1	0.2307 (2)	0.61487 (14)	0.60279 (9)	0.0324 (3)	
C2	0.24256 (19)	0.49996 (14)	0.56282 (9)	0.0323 (3)	
C3	0.2414 (2)	0.37946 (15)	0.58555 (10)	0.0375 (4)	
H3	0.2332	0.3559	0.6387	0.045*	
C4	0.2532 (2)	0.29710 (17)	0.52459 (11)	0.0437 (4)	
C5	0.2648 (2)	0.32726 (19)	0.44430 (11)	0.0491 (5)	
H5	0.2715	0.2671	0.4059	0.059*	
C6	0.2664 (2)	0.44598 (18)	0.42199 (10)	0.0456 (4)	
H6	0.2745	0.4688	0.3687	0.055*	
C7	0.2553 (2)	0.53018 (16)	0.48228 (9)	0.0369 (4)	
C8	0.2376 (2)	0.70287 (16)	0.54652 (10)	0.0391 (4)	
C9	0.2333 (3)	0.83607 (17)	0.54796 (12)	0.0547 (5)	
H9A	0.2265	0.8632	0.6024	0.082*	
H9B	0.1259	0.8644	0.5101	0.082*	
H9C	0.3449	0.8671	0.5324	0.082*	
C10	0.4518 (2)	0.63802 (13)	0.75603 (9)	0.0298 (3)	
C11	0.5530 (2)	0.74450 (14)	0.75670 (10)	0.0375 (3)	
H11	0.4957	0.8143	0.7332	0.045*	

C12	0.7390 (2)	0.74468 (16)	0.79277 (11)	0.0420 (4)	0.887 (3)
H12	0.8083	0.8150	0.7925	0.050*	0.887 (3)
C12'	0.7390 (2)	0.74468 (16)	0.79277 (11)	0.0420 (4)	0.11
F2'	0.8472 (11)	0.8379 (6)	0.8099 (5)	0.042 (3)	0.113 (3)
C13	0.8244 (2)	0.64277 (16)	0.82925 (10)	0.0409 (4)	
H13	0.9504	0.6434	0.8535	0.049*	
C14	0.7198 (2)	0.54100 (15)	0.82885 (10)	0.0402 (4)	0.887 (3)
F2	0.80371 (19)	0.44337 (11)	0.86633 (8)	0.0619 (4)	0.887 (3)
C14'	0.7198 (2)	0.54100 (15)	0.82885 (10)	0.0402 (4)	0.11
H14'	0.7770	0.4726	0.8546	0.048*	0.113 (3)
C15	0.5338 (2)	0.53388 (14)	0.79236 (9)	0.0354 (3)	
H15	0.4666	0.4626	0.7921	0.042*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0314 (2)	0.0312 (2)	0.0326 (2)	0.00158 (14)	0.01275 (15)	-0.00048 (14)
F1	0.0746 (8)	0.0435 (6)	0.0622 (7)	0.0073 (6)	-0.0001 (6)	-0.0119 (5)
O1	0.0432 (7)	0.0576 (8)	0.0303 (6)	-0.0037 (5)	0.0070 (5)	0.0065 (5)
O2	0.0380 (6)	0.0387 (6)	0.0384 (6)	-0.0052 (5)	0.0158 (5)	-0.0007 (5)
O3	0.0392 (6)	0.0379 (6)	0.0478 (7)	0.0076 (5)	0.0157 (5)	-0.0022 (5)
C1	0.0281 (7)	0.0384 (8)	0.0318 (7)	0.0015 (6)	0.0084 (6)	0.0008 (6)
C2	0.0227 (7)	0.0446 (9)	0.0293 (7)	0.0015 (6)	0.0044 (5)	-0.0022 (6)
C3	0.0337 (8)	0.0431 (9)	0.0347 (8)	0.0026 (6)	0.0040 (6)	-0.0033 (6)
C4	0.0373 (9)	0.0446 (10)	0.0459 (9)	0.0041 (7)	-0.0007 (7)	-0.0090 (7)
C5	0.0390 (9)	0.0659 (13)	0.0404 (9)	0.0022 (8)	0.0023 (7)	-0.0205 (9)
C6	0.0346 (8)	0.0720 (13)	0.0296 (8)	-0.0023 (8)	0.0046 (6)	-0.0066 (8)
C7	0.0265 (7)	0.0530 (10)	0.0306 (7)	-0.0012 (6)	0.0044 (6)	0.0004 (7)
C8	0.0342 (8)	0.0475 (10)	0.0360 (8)	0.0008 (7)	0.0072 (6)	0.0050 (7)
C9	0.0671 (13)	0.0459 (11)	0.0512 (11)	-0.0017 (9)	0.0116 (9)	0.0113 (8)
C10	0.0329 (7)	0.0301 (7)	0.0287 (7)	0.0007 (6)	0.0117 (6)	-0.0034 (5)
C11	0.0417 (8)	0.0307 (8)	0.0419 (8)	-0.0004 (6)	0.0126 (7)	0.0003 (6)
C12	0.0406 (9)	0.0416 (9)	0.0457 (9)	-0.0081 (7)	0.0131 (7)	-0.0085 (7)
C12'	0.0406 (9)	0.0416 (9)	0.0457 (9)	-0.0081 (7)	0.0131 (7)	-0.0085 (7)
F2'	0.038 (4)	0.041 (5)	0.048 (5)	-0.014 (3)	0.013 (3)	-0.005 (3)
C13	0.0344 (8)	0.0540 (10)	0.0345 (8)	0.0003 (7)	0.0072 (6)	-0.0090 (7)
C14	0.0445 (9)	0.0409 (9)	0.0348 (8)	0.0089 (7)	0.0065 (7)	0.0014 (7)
F2	0.0601 (8)	0.0469 (8)	0.0715 (9)	0.0124 (6)	-0.0070 (7)	0.0121 (6)
C14'	0.0445 (9)	0.0409 (9)	0.0348 (8)	0.0089 (7)	0.0065 (7)	0.0014 (7)
C15	0.0416 (8)	0.0303 (8)	0.0355 (8)	-0.0009 (6)	0.0103 (6)	-0.0006 (6)

Geometric parameters (Å, °)

S1—O3	1.4339 (11)	C6—H6	0.9300
S1—O2	1.4349 (11)	C8—C9	1.479 (2)
S1—C1	1.7354 (15)	C9—H9A	0.9600
S1—C10	1.7627 (16)	C9—H9B	0.9600
F1—C4	1.361 (2)	C9—H9C	0.9600

O1—C8	1.366 (2)	C10—C15	1.386 (2)
O1—C7	1.374 (2)	C10—C11	1.391 (2)
C1—C8	1.357 (2)	C11—C12	1.374 (2)
C1—C2	1.447 (2)	C11—H11	0.9300
C2—C3	1.390 (2)	C12—C13	1.375 (2)
C2—C7	1.395 (2)	C12—H12	0.9300
C3—C4	1.376 (2)	C13—C14	1.361 (2)
C3—H3	0.9300	C13—H13	0.9300
C4—C5	1.389 (3)	C14—F2	1.3383 (19)
C5—C6	1.369 (3)	C14—C15	1.378 (2)
C5—H5	0.9300	C15—H15	0.9300
C6—C7	1.381 (2)		
O3—S1—O2	119.64 (7)	C1—C8—O1	110.14 (15)
O3—S1—C1	108.82 (7)	C1—C8—C9	134.79 (16)
O2—S1—C1	107.65 (7)	O1—C8—C9	115.07 (15)
O3—S1—C10	107.83 (7)	C8—C9—H9A	109.5
O2—S1—C10	107.96 (7)	C8—C9—H9B	109.5
C1—S1—C10	103.84 (7)	H9A—C9—H9B	109.5
C8—O1—C7	107.34 (12)	C8—C9—H9C	109.5
C8—C1—C2	107.87 (13)	H9A—C9—H9C	109.5
C8—C1—S1	126.82 (13)	H9B—C9—H9C	109.5
C2—C1—S1	125.29 (11)	C15—C10—C11	121.37 (15)
C3—C2—C7	119.67 (15)	C15—C10—S1	119.32 (12)
C3—C2—C1	136.08 (14)	C11—C10—S1	119.29 (12)
C7—C2—C1	104.24 (14)	C12—C11—C10	118.79 (15)
C4—C3—C2	115.85 (15)	C12—C11—H11	120.6
C4—C3—H3	122.1	C10—C11—H11	120.6
C2—C3—H3	122.1	C11—C12—C13	121.22 (16)
F1—C4—C3	117.79 (16)	C11—C12—H12	119.4
F1—C4—C5	117.77 (16)	C13—C12—H12	119.4
C3—C4—C5	124.43 (18)	C14—C13—C12	118.25 (16)
C6—C5—C4	119.70 (16)	C14—C13—H13	120.9
C6—C5—H5	120.2	C12—C13—H13	120.9
C4—C5—H5	120.2	F2—C14—C13	117.58 (16)
C5—C6—C7	116.84 (16)	F2—C14—C15	118.89 (16)
C5—C6—H6	121.6	C13—C14—C15	123.53 (15)
C7—C6—H6	121.6	C14—C15—C10	116.82 (15)
O1—C7—C6	126.08 (15)	C14—C15—H15	121.6
O1—C7—C2	110.41 (14)	C10—C15—H15	121.6
C6—C7—C2	123.51 (17)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C5—H5 \cdots F2 ⁱ	0.93	2.45	3.302 (2)	153

C15—H15 \cdots O3 ⁱⁱ	0.93	2.47	3.3754 (19)	165
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Symmetry codes: (i) $x-1/2, -y+1/2, z-1/2$; (ii) $-x+1/2, y-1/2, -z+3/2$.