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

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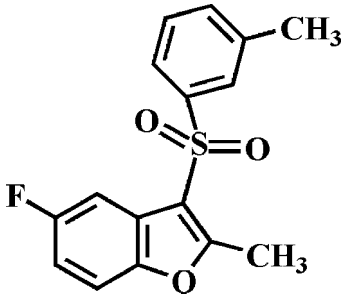
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.037; wR factor = 0.103; data-to-parameter ratio = 17.0.

In the title compound, $C_{16}H_{13}FO_3S$, the dihedral angle between the mean planes of the benzofuran ring system and the 3-methylphenyl ring is 80.96 (4)°. In the crystal, molecules are linked *via* pairs of π–π interactions between furan and benzene rings, with centroid–centroid distances of 3.758 (1) and 3.771 (1) Å. A similar interaction is found between furan rings, with a centroid–centroid distance of 3.661 (1) Å between neighbouring molecules. The molecules stack along the *a*-axis direction. In addition, C—H···O and C—H···π hydrogen bonds are observed between inversion-related dimers.

Related literature

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



Experimental

Crystal data

$C_{16}H_{13}FO_3S$
M_r = 304.32
Triclinic, $P\overline{1}$
a = 7.4406 (1) Å
b = 9.1291 (2) Å
c = 11.2073 (2) Å
α = 82.891 (1)°
β = 73.301 (1)°
γ = 77.613 (1)°
V = 710.62 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 173 K
0.37 × 0.30 × 0.28 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.695, T_{max} = 0.746
12591 measured reflections
3263 independent reflections
2868 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.103
S = 1.07
3263 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

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

*Cg*3 is the centroid of the C10–C15 3-methylphenyl ring.

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11···O2^{i}	0.95	2.51	3.450 (2)	172
C6—H6···*Cg*3^{ii}	0.95	2.76	3.556 (2)	142

Symmetry codes: (i) $-x+2, -y, -z+1$; (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 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: BG2523).

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supporting information

Acta Cryst. (2014). E**70**, o482 [doi:10.1107/S1600536814006321]

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S1. Experimental

S1.1. Synthesis and crystallization

3-Chloroperoxybenzoic acid (77%, 448 mg, 2.0 mmol) was added in small portions to a stirred solution of 5-fluoro-2-methyl-3-(3-methylphenylsulfanyl)-1-benzofuran (245 mg, 0.9 mmol) in dichloromethane (30 mL) at 273 K. After being stirred at room temperature for 8h, the mixture was washed with a 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, 4:1 *v*/*v*) to afford the title compound as a colorless solid [yield 71%, m.p. 375–376 K; R_f = 0.51 (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 diisopropyl ether, at room temperature.

S1.2. 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).

S2. Results and discussion

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

In the title molecule (Fig. 1), the benzofuran ring system is essentially planar, with a mean deviation of 0.006 (1) Å from the least-squares plane defined by the nine constituent atoms. The 3-methylphenyl ring is essentially planar, with a mean deviation of 0.003 (1) Å from the least-squares plane defined by the six constituent atoms. The dihedral angle formed by the benzofuran ring system and the 3-methylphenyl ring is 80.96 (4)°.

In the crystal structure (Fig. 2), the molecules are linked *via* pairs of $\pi\cdots\pi$ interactions between the furan and benzene rings, and between the furan rings of neighbouring molecules. The molecules stack along the *a*-axis direction. The relevant centroid names for $\pi\cdots\pi$ stacking interactions are Cg1 for the furan ring (C1/C2/C7/O1/O8) and Cg2 for the benzene ring (C2–C7). The centroid-centroid separations of Cg1$\cdots$Cg2^{ii} [(ii): - *x* + 1, - *y* + 1, - *z* + 1], Cg1$\cdots$Cg2^{iii} and Cg1$\cdots$Cg1^{iii} [(iii): - *x* + 2,- *y* + 1, - *z* + 1] are 3.758 (1), 3.771 (1) and 3.661 (1) Å, respectively. The slippages of Cg1$\cdots$Cg2^{ii}, Cg1$\cdots$Cg2^{iii} and Cg1$\cdots$Cg1^{iii} are 1.227 (1), 1.266 (1) Å and 0.887 (1) Å, respectively. In the crystal packing (Fig. 2), intermolecular C—H$\cdots$O and C—H$\cdots\pi$ hydrogen bonds are observed between inversion-related dimers.

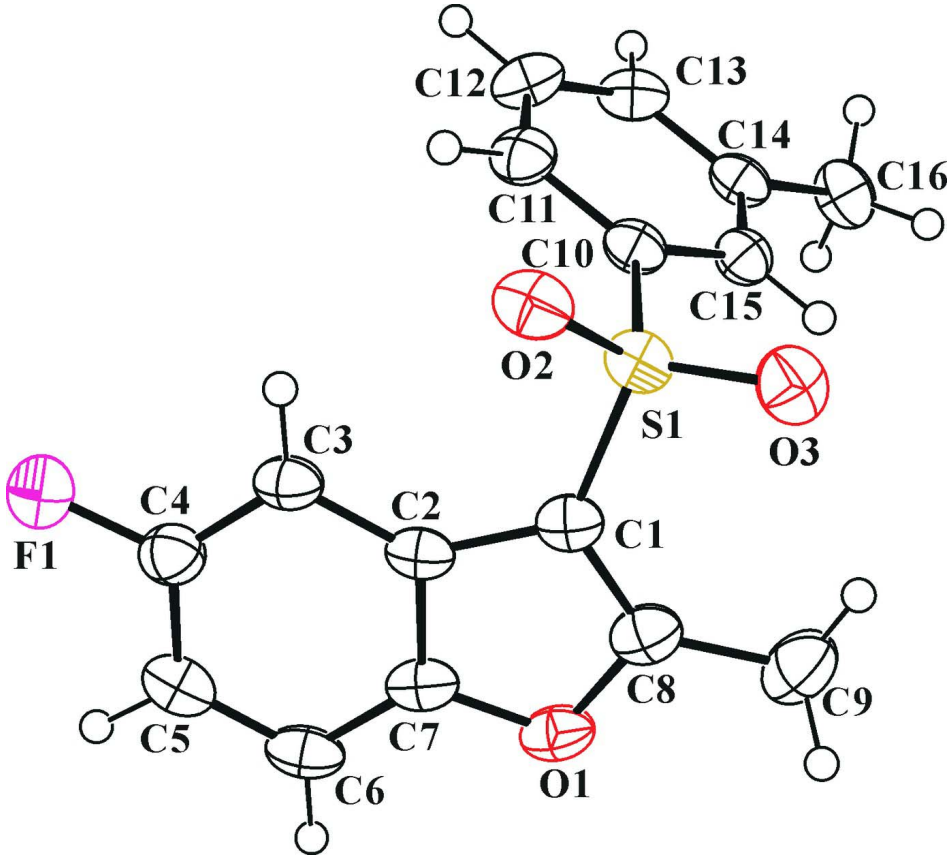

Figure 1

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.

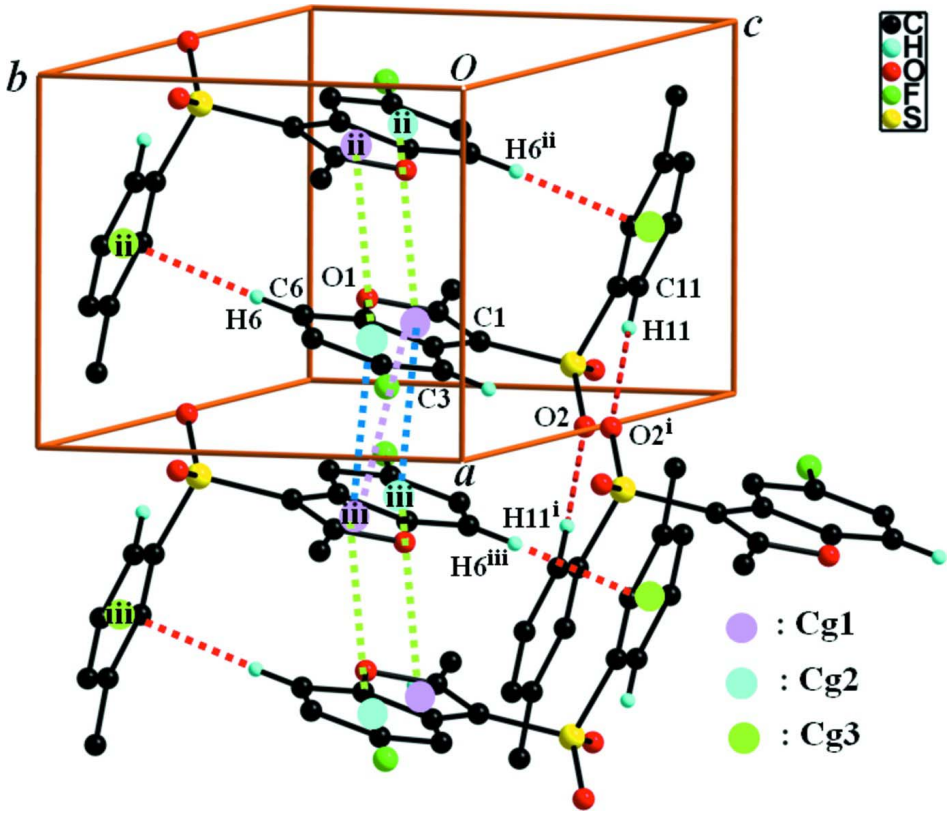

Figure 2

A view of the C—H..O, C—H···π 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, - z + 1 ; (ii) - x + 1,- y + 1,- z + 1; (iii)- x + 2,- y + 1, - z + 1

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Crystal data

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Hall symbol: -P 1
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c = 11.2073 (2) Å
α = 82.891 (1)°
β = 73.301 (1)°
γ = 77.613 (1)°
V = 710.62 (2) Å^3
Z = 2
$F(000)$ = 316
D_x = 1.422 Mg m^{-3}
Melting point = 375–376 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5506 reflections
θ = 2.9–27.5°
μ = 0.25 mm^{-1}
T = 173 K
Block, colourless
0.37 × 0.30 × 0.28 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} = 0.695, T_{max} = 0.746
12591 measured reflections
3263 independent reflections
2868 reflections with $I > 2\sigma(I)$

R_{int} = 0.026
θ_{max} = 27.5°, θ_{min} = 1.9°
h = −9→9
k = −11→11
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.103
S = 1.07
3263 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
$w = 1/[\sigma^2(F_o^2) + (0.056P)^2 + 0.1689P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 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	0.87603 (5)	0.18979 (4)	0.70443 (3)	0.03242 (12)
F1	0.84714 (17)	0.30138 (12)	0.19261 (9)	0.0556 (3)
O1	0.69505 (15)	0.60973 (11)	0.60126 (11)	0.0391 (3)
O2	1.02286 (15)	0.10305 (12)	0.61294 (11)	0.0395 (3)
O3	0.91181 (16)	0.21354 (13)	0.81879 (11)	0.0432 (3)
C1	0.80671 (19)	0.36430 (15)	0.63233 (14)	0.0310 (3)
C2	0.79931 (18)	0.39130 (15)	0.50423 (14)	0.0295 (3)
C3	0.8409 (2)	0.30421 (15)	0.40267 (14)	0.0338 (3)
H3	0.8885	0.1990	0.4076	0.041*
C4	0.8087 (2)	0.38018 (17)	0.29498 (15)	0.0382 (3)
C5	0.7414 (2)	0.53393 (18)	0.28163 (16)	0.0404 (4)
H5	0.7240	0.5792	0.2037	0.048*
C6	0.7004 (2)	0.61985 (16)	0.38156 (16)	0.0389 (4)
H6	0.6545	0.7252	0.3756	0.047*
C7	0.7292 (2)	0.54522 (15)	0.49103 (15)	0.0333 (3)
C8	0.7415 (2)	0.49761 (17)	0.68625 (15)	0.0371 (3)
C9	0.7072 (3)	0.5457 (2)	0.81330 (18)	0.0531 (5)
H9A	0.5697	0.5784	0.8494	0.080*
H9B	0.7729	0.6292	0.8091	0.080*
H9C	0.7562	0.4613	0.8656	0.080*
C10	0.6704 (2)	0.10864 (15)	0.74169 (13)	0.0300 (3)
C11	0.6516 (2)	0.01107 (16)	0.66260 (14)	0.0354 (3)

H11	0.7496	−0.0155	0.5888	0.042*
C12	0.4846 (2)	−0.04647 (17)	0.69504 (16)	0.0404 (4)
H12	0.4678	−0.1138	0.6427	0.048*
C13	0.3430 (2)	−0.00721 (16)	0.80208 (16)	0.0373 (3)
H13	0.2298	−0.0480	0.8220	0.045*
C14	0.3615 (2)	0.09077 (15)	0.88184 (14)	0.0319 (3)
C15	0.5287 (2)	0.14767 (16)	0.85034 (13)	0.0311 (3)
H15	0.5465	0.2137	0.9034	0.037*
C16	0.2048 (2)	0.13571 (19)	0.99669 (14)	0.0414 (4)
H16A	0.1115	0.2202	0.9746	0.062*
H16B	0.2589	0.1658	1.0579	0.062*
H16C	0.1415	0.0505	1.0327	0.062*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.02348 (19)	0.0338 (2)	0.0379 (2)	−0.00384 (14)	−0.00764 (15)	0.00143 (14)
F1	0.0765 (8)	0.0477 (6)	0.0407 (5)	−0.0112 (5)	−0.0125 (5)	−0.0050 (4)
O1	0.0349 (6)	0.0270 (5)	0.0518 (6)	−0.0042 (4)	−0.0050 (5)	−0.0076 (4)
O2	0.0261 (5)	0.0355 (5)	0.0482 (6)	0.0016 (4)	−0.0033 (5)	0.0008 (5)
O3	0.0350 (6)	0.0538 (7)	0.0446 (6)	−0.0116 (5)	−0.0163 (5)	0.0018 (5)
C1	0.0224 (6)	0.0298 (7)	0.0390 (7)	−0.0061 (5)	−0.0045 (6)	−0.0022 (5)
C2	0.0202 (6)	0.0248 (6)	0.0407 (7)	−0.0046 (5)	−0.0047 (6)	0.0014 (5)
C3	0.0300 (7)	0.0251 (6)	0.0421 (8)	−0.0036 (5)	−0.0050 (6)	−0.0009 (6)
C4	0.0370 (8)	0.0356 (7)	0.0403 (8)	−0.0089 (6)	−0.0062 (7)	−0.0025 (6)
C5	0.0354 (8)	0.0374 (8)	0.0460 (9)	−0.0085 (6)	−0.0112 (7)	0.0091 (7)
C6	0.0309 (8)	0.0252 (7)	0.0568 (10)	−0.0034 (6)	−0.0106 (7)	0.0058 (6)
C7	0.0230 (7)	0.0253 (6)	0.0478 (8)	−0.0043 (5)	−0.0034 (6)	−0.0036 (6)
C8	0.0279 (7)	0.0357 (7)	0.0464 (8)	−0.0093 (6)	−0.0042 (6)	−0.0065 (6)
C9	0.0540 (11)	0.0512 (10)	0.0535 (10)	−0.0120 (8)	−0.0058 (9)	−0.0193 (8)
C10	0.0257 (7)	0.0277 (6)	0.0346 (7)	−0.0026 (5)	−0.0089 (6)	0.0033 (5)
C11	0.0317 (7)	0.0290 (7)	0.0405 (8)	0.0006 (6)	−0.0056 (6)	−0.0045 (6)
C12	0.0374 (8)	0.0281 (7)	0.0556 (10)	−0.0032 (6)	−0.0113 (7)	−0.0110 (6)
C13	0.0303 (7)	0.0280 (7)	0.0519 (9)	−0.0065 (6)	−0.0093 (7)	0.0009 (6)
C14	0.0282 (7)	0.0289 (7)	0.0354 (7)	−0.0022 (5)	−0.0090 (6)	0.0059 (5)
C15	0.0295 (7)	0.0311 (7)	0.0319 (7)	−0.0031 (5)	−0.0103 (6)	0.0008 (5)
C16	0.0314 (8)	0.0525 (9)	0.0358 (8)	−0.0078 (7)	−0.0045 (6)	0.0025 (7)

Geometric parameters (Å, º)

S1—O3	1.4319 (12)	C8—C9	1.478 (2)
S1—O2	1.4346 (11)	C9—H9A	0.9800
S1—C1	1.7394 (14)	C9—H9B	0.9800
S1—C10	1.7626 (14)	C9—H9C	0.9800
F1—C4	1.3575 (18)	C10—C11	1.385 (2)
O1—C8	1.368 (2)	C10—C15	1.389 (2)
O1—C7	1.3686 (18)	C11—C12	1.387 (2)
C1—C8	1.357 (2)	C11—H11	0.9500

C1—C2	1.441 (2)	C12—C13	1.377 (2)
C2—C3	1.393 (2)	C12—H12	0.9500
C2—C7	1.3954 (19)	C13—C14	1.393 (2)
C3—C4	1.372 (2)	C13—H13	0.9500
C3—H3	0.9500	C14—C15	1.386 (2)
C4—C5	1.390 (2)	C14—C16	1.499 (2)
C5—C6	1.371 (2)	C15—H15	0.9500
C5—H5	0.9500	C16—H16A	0.9800
C6—C7	1.377 (2)	C16—H16B	0.9800
C6—H6	0.9500	C16—H16C	0.9800

O3—S1—O2	119.73 (7)	C8—C9—H9A	109.5
O3—S1—C1	108.28 (7)	C8—C9—H9B	109.5
O2—S1—C1	107.74 (7)	H9A—C9—H9B	109.5
O3—S1—C10	108.10 (7)	C8—C9—H9C	109.5
O2—S1—C10	108.25 (7)	H9A—C9—H9C	109.5
C1—S1—C10	103.57 (6)	H9B—C9—H9C	109.5
C8—O1—C7	107.14 (11)	C11—C10—C15	121.75 (13)
C8—C1—C2	107.50 (13)	C11—C10—S1	120.15 (11)
C8—C1—S1	127.01 (12)	C15—C10—S1	118.09 (11)
C2—C1—S1	125.42 (11)	C10—C11—C12	117.64 (14)
C3—C2—C7	119.40 (14)	C10—C11—H11	121.2
C3—C2—C1	136.00 (13)	C12—C11—H11	121.2
C7—C2—C1	104.60 (13)	C13—C12—C11	120.88 (14)
C4—C3—C2	115.65 (13)	C13—C12—H12	119.6
C4—C3—H3	122.2	C11—C12—H12	119.6
C2—C3—H3	122.2	C12—C13—C14	121.61 (14)
F1—C4—C3	118.38 (14)	C12—C13—H13	119.2
F1—C4—C5	116.81 (15)	C14—C13—H13	119.2
C3—C4—C5	124.80 (15)	C15—C14—C13	117.73 (14)
C6—C5—C4	119.62 (15)	C15—C14—C16	120.97 (13)
C6—C5—H5	120.2	C13—C14—C16	121.30 (14)
C4—C5—H5	120.2	C14—C15—C10	120.38 (13)
C5—C6—C7	116.43 (13)	C14—C15—H15	119.8
C5—C6—H6	121.8	C10—C15—H15	119.8
C7—C6—H6	121.8	C14—C16—H16A	109.5
O1—C7—C6	125.55 (13)	C14—C16—H16B	109.5
O1—C7—C2	110.36 (13)	H16A—C16—H16B	109.5
C6—C7—C2	124.09 (14)	C14—C16—H16C	109.5
C1—C8—O1	110.40 (14)	H16A—C16—H16C	109.5
C1—C8—C9	134.70 (16)	H16B—C16—H16C	109.5
O1—C8—C9	114.88 (14)		

O3—S1—C1—C8	−18.19 (15)	C1—C2—C7—C6	179.58 (13)
O2—S1—C1—C8	−149.03 (13)	C2—C1—C8—O1	−0.94 (16)
C10—S1—C1—C8	96.42 (14)	S1—C1—C8—O1	−178.00 (10)
O3—S1—C1—C2	165.25 (11)	C2—C1—C8—C9	177.59 (17)
O2—S1—C1—C2	34.41 (13)	S1—C1—C8—C9	0.5 (3)

C10—S1—C1—C2	−80.14 (13)	C7—O1—C8—C1	0.89 (16)
C8—C1—C2—C3	−178.42 (16)	C7—O1—C8—C9	−177.96 (13)
S1—C1—C2—C3	−1.3 (2)	O3—S1—C10—C11	−149.08 (12)
C8—C1—C2—C7	0.61 (15)	O2—S1—C10—C11	−18.00 (14)
S1—C1—C2—C7	177.72 (10)	C1—S1—C10—C11	96.18 (13)
C7—C2—C3—C4	0.2 (2)	O3—S1—C10—C15	32.04 (13)
C1—C2—C3—C4	179.12 (15)	O2—S1—C10—C15	163.13 (11)
C2—C3—C4—F1	179.80 (13)	C1—S1—C10—C15	−82.69 (12)
C2—C3—C4—C5	0.8 (2)	C15—C10—C11—C12	0.4 (2)
F1—C4—C5—C6	−179.85 (14)	S1—C10—C11—C12	−178.41 (11)
C3—C4—C5—C6	−0.8 (3)	C10—C11—C12—C13	0.1 (2)
C4—C5—C6—C7	−0.2 (2)	C11—C12—C13—C14	−0.2 (2)
C8—O1—C7—C6	179.87 (14)	C12—C13—C14—C15	−0.3 (2)
C8—O1—C7—C2	−0.49 (15)	C12—C13—C14—C16	178.60 (14)
C5—C6—C7—O1	−179.25 (14)	C13—C14—C15—C10	0.8 (2)
C5—C6—C7—C2	1.2 (2)	C16—C14—C15—C10	−178.06 (13)
C3—C2—C7—O1	179.16 (12)	C11—C10—C15—C14	−0.9 (2)
C1—C2—C7—O1	−0.07 (15)	S1—C10—C15—C14	177.93 (10)
C3—C2—C7—C6	−1.2 (2)		

Hydrogen-bond geometry (Å, º)

Cg3 is the centroid of the C10–C15 3-methylphenyl ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···O2^{i}	0.95	2.51	3.450 (2)	172
C6—H6···*Cg*3^{ii}	0.95	2.76	3.556 (2)	142

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