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5-Bromo-3-(4-fluorophenylsulfonyl)-2,7-dimethyl-1-benzofuran

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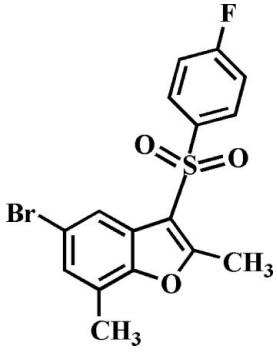
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.028; wR factor = 0.074; data-to-parameter ratio = 18.3.

In the title compound, $C_{16}H_{12}BrFO_3S$, the 4-fluorophenyl ring makes a dihedral angle of 72.35 (5)° with the mean plane [r.m.s. deviation = 0.008 (1) Å] of the benzofuran fragment. In the crystal, molecules are linked into [010] chains *via* two different inversion-generated pairs of C—H···O hydrogen bonds. The crystal structure also exhibits slipped π–π interactions between the benzene rings of neighbouring molecules [centroid–centroid distance = 3.667 (2) Å and slippage = 1.341 (2) Å], and between the benzene and the furan rings of neighbouring molecules [centroid–centroid distance = 3.759 (2) Å and slippage = 0.757 (2) Å].

Related literature

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



Experimental

Crystal data

$C_{16}H_{12}BrFO_3S$
M_r = 383.23
Triclinic, $P\overline{1}$
a = 8.0789 (1) Å
b = 9.2754 (2) Å
c = 11.3555 (2) Å
α = 71.283 (1)°
β = 72.645 (1)°
γ = 70.042 (1)°
V = 740.30 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.94 mm^{-1}
T = 173 K
0.29 × 0.21 × 0.18 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.527, T_{max} = 0.746
14090 measured reflections
3680 independent reflections
3259 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.074
S = 1.02
3680 reflections
201 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 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	2.53	3.408 (2)	154
C9—H9A···O2^{ii}	0.98	2.50	3.304 (2)	139

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

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6977).

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supporting information

Acta Cryst. (2012). E**68**, o3208 [doi:10.1107/S1600536812043607]

5-Bromo-3-(4-fluorophenylsulfonyl)-2,7-dimethyl-1-benzofuran

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S1. Comment

As a part of our continuing study of 5-bromo-2-methyl-1-benzofuran derivatives containing 4-fluorophenylsulfonyl (Choi *et al.*, 2010) and 4-methylphenylsulfonyl (Choi *et al.*, 2012) substituents in 3-position, 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 0.008 (1) Å from the least-squares plane defined by the nine constituent atoms. The dihedral angle between the 4-fluorophenyl ring and the mean plane of the benzofuran ring is 72.35 (5). In the crystal structure (Fig. 2), molecules are linked *via* pairs of C—H···O hydrogen bonds (Table 1), forming inversion dimers. The crystal packing (Fig. 2) also exhibits slipped π–π interactions; the first one between the benzene rings of neighbouring molecules, with a Cg1···Cg1^{iii} distance of 3.667 (2) Å and an interplanar distance of 3.413 (2) Å resulting in a slippage of 1.341 (2) Å (Cg1 is the centroid of the C2–C7 benzene ring), and the second one between the benzene and the furan rings of neighbouring molecules, with a Cg1···Cg2^{ii} distance of 3.759 (2) Å and an interplanar distance of 3.682 (2) Å resulting in a slippage of 0.757 (2) Å (Cg2 is the C1/C2/C7/O1/C8 furan ring).

S2. Experimental

3-Chloroperoxybenzoic acid (77%, 381 mg, 1.7 mmol) was added in small portions to a stirred solution of 5-bromo-3-(4-fluorophenylsulfanyl)-2,7-dimethyl-1-benzofuran (281 mg, 0.8 mmol) in dichloromethane (40 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 (hexane–ethyl acetate, 4:1 v/v) to afford the title compound as a colorless solid [yield 71%, m.p. 472–473 K; R_f = 0.57 (hexane–ethyl acetate, 4:1 v/v)]. Colourless blocks 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. The positions of methyl hydrogens were optimized rotationally.

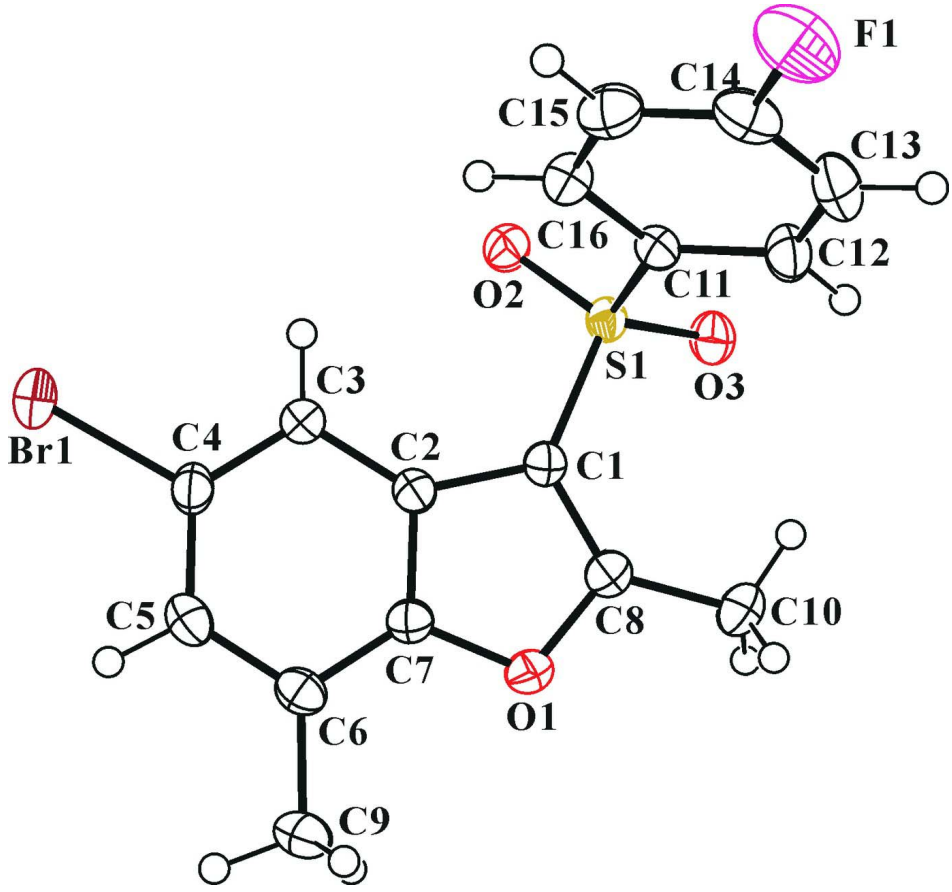

Figure 1

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.

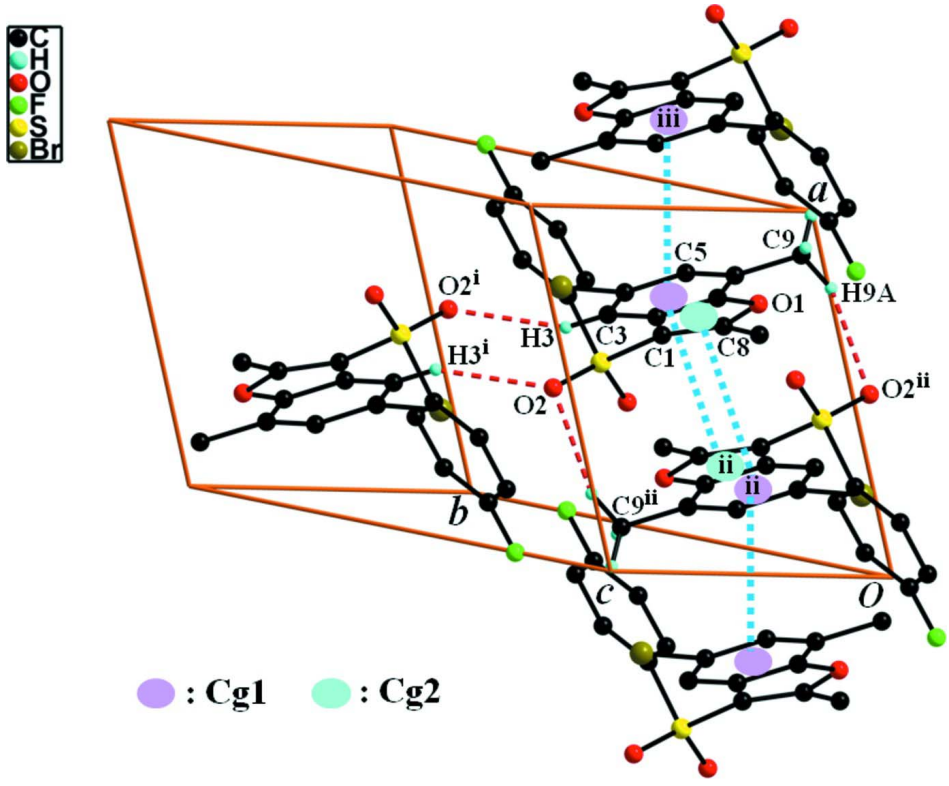

Figure 2

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

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$C_{16}H_{12}BrFO_3S$
M_r = 383.23
Triclinic, $P\overline{1}$
Hall symbol: -P 1
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c = 11.3555 (2) Å
α = 71.283 (1)°
β = 72.645 (1)°
γ = 70.042 (1)°
V = 740.30 (2) Å^3

Z = 2
F(000) = 384
D_x = 1.719 Mg m^{-3}
Melting point = 472–473 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6655 reflections
θ = 2.7–28.3°
μ = 2.94 mm^{-1}
T = 173 K
Block, colourless
0.29 × 0.21 × 0.18 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} = 0.527, T_{max} = 0.746
14090 measured reflections
3680 independent reflections
3259 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

θ_{max} = 28.3°, θ_{min} = 1.9°
h = −10→10
k = −12→12
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.074
S = 1.02
3680 reflections
201 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
$w = 1/[\sigma^2(F_o^2) + (0.0417P)^2 + 0.237P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 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}
Br1	0.75349 (3)	0.10966 (3)	0.797351 (17)	0.03912 (8)
S1	0.48725 (5)	0.40486 (5)	0.29104 (4)	0.02342 (10)
F1	1.00247 (19)	0.77090 (18)	0.00357 (14)	0.0571 (4)
O1	0.75109 (17)	−0.04433 (15)	0.32764 (12)	0.0274 (3)
O2	0.40458 (16)	0.45572 (16)	0.40738 (11)	0.0286 (3)
O3	0.37705 (17)	0.41993 (16)	0.20642 (12)	0.0315 (3)
C1	0.6075 (2)	0.2081 (2)	0.33360 (16)	0.0242 (3)
C2	0.6733 (2)	0.1303 (2)	0.45008 (15)	0.0227 (3)
C3	0.6674 (2)	0.1745 (2)	0.55829 (16)	0.0251 (3)
H3	0.6078	0.2785	0.5692	0.030*
C4	0.7538 (2)	0.0574 (2)	0.64843 (16)	0.0271 (3)
C5	0.8430 (2)	−0.0963 (2)	0.63592 (17)	0.0285 (4)
H5	0.9010	−0.1707	0.7011	0.034*
C6	0.8486 (2)	−0.1426 (2)	0.52969 (17)	0.0268 (3)
C7	0.7611 (2)	−0.0248 (2)	0.44028 (16)	0.0243 (3)
C8	0.6581 (2)	0.0987 (2)	0.26428 (16)	0.0265 (3)
C9	0.9424 (3)	−0.3065 (2)	0.51205 (19)	0.0331 (4)
H9A	0.8612	−0.3460	0.4891	0.050*
H9B	0.9767	−0.3761	0.5915	0.050*
H9C	1.0506	−0.3045	0.4440	0.050*
C10	0.6362 (3)	0.1018 (3)	0.13857 (18)	0.0346 (4)
H10A	0.5784	0.2103	0.0967	0.052*
H10B	0.5610	0.0329	0.1499	0.052*

H10C	0.7547	0.0644	0.0858	0.052*
C11	0.6478 (2)	0.5107 (2)	0.20442 (16)	0.0244 (3)
C12	0.6821 (3)	0.5500 (2)	0.07255 (17)	0.0321 (4)
H12	0.6237	0.5157	0.0295	0.038*
C13	0.8014 (3)	0.6392 (3)	0.00442 (19)	0.0394 (5)
H13	0.8256	0.6685	−0.0859	0.047*
C14	0.8840 (3)	0.6845 (2)	0.0705 (2)	0.0376 (4)
C15	0.8550 (3)	0.6459 (3)	0.2007 (2)	0.0370 (4)
H15	0.9169	0.6782	0.2427	0.044*
C16	0.7330 (2)	0.5585 (2)	0.26933 (17)	0.0300 (4)
H16	0.7078	0.5316	0.3597	0.036*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.05355 (14)	0.04150 (14)	0.02493 (11)	−0.01030 (10)	−0.01583 (8)	−0.00744 (8)
S1	0.02474 (19)	0.0241 (2)	0.02165 (19)	−0.00430 (16)	−0.00808 (15)	−0.00546 (16)
F1	0.0558 (8)	0.0497 (9)	0.0620 (9)	−0.0322 (7)	−0.0040 (7)	0.0026 (7)
O1	0.0325 (6)	0.0237 (6)	0.0290 (6)	−0.0070 (5)	−0.0088 (5)	−0.0090 (5)
O2	0.0296 (6)	0.0290 (7)	0.0250 (6)	−0.0048 (5)	−0.0041 (5)	−0.0089 (5)
O3	0.0325 (6)	0.0346 (7)	0.0306 (6)	−0.0073 (6)	−0.0154 (5)	−0.0061 (6)
C1	0.0276 (8)	0.0232 (8)	0.0228 (7)	−0.0067 (7)	−0.0077 (6)	−0.0049 (7)
C2	0.0230 (7)	0.0222 (8)	0.0226 (7)	−0.0064 (6)	−0.0058 (6)	−0.0041 (6)
C3	0.0285 (8)	0.0234 (9)	0.0237 (8)	−0.0065 (7)	−0.0063 (6)	−0.0059 (7)
C4	0.0319 (8)	0.0303 (9)	0.0201 (7)	−0.0097 (7)	−0.0074 (6)	−0.0045 (7)
C5	0.0305 (8)	0.0260 (9)	0.0265 (8)	−0.0090 (7)	−0.0089 (7)	0.0009 (7)
C6	0.0271 (8)	0.0224 (9)	0.0294 (8)	−0.0091 (7)	−0.0058 (7)	−0.0020 (7)
C7	0.0262 (8)	0.0226 (9)	0.0253 (8)	−0.0075 (7)	−0.0055 (6)	−0.0061 (7)
C8	0.0277 (8)	0.0264 (9)	0.0267 (8)	−0.0077 (7)	−0.0062 (6)	−0.0075 (7)
C9	0.0357 (9)	0.0217 (9)	0.0397 (10)	−0.0056 (8)	−0.0106 (8)	−0.0047 (8)
C10	0.0431 (10)	0.0364 (11)	0.0302 (9)	−0.0093 (9)	−0.0125 (8)	−0.0132 (8)
C11	0.0267 (8)	0.0208 (8)	0.0235 (8)	−0.0034 (7)	−0.0072 (6)	−0.0041 (7)
C12	0.0360 (9)	0.0344 (10)	0.0253 (8)	−0.0077 (8)	−0.0113 (7)	−0.0041 (8)
C13	0.0435 (11)	0.0415 (12)	0.0254 (9)	−0.0122 (9)	−0.0071 (8)	0.0027 (8)
C14	0.0367 (10)	0.0264 (10)	0.0437 (11)	−0.0116 (8)	−0.0042 (8)	−0.0013 (9)
C15	0.0387 (10)	0.0345 (11)	0.0445 (11)	−0.0135 (9)	−0.0100 (8)	−0.0135 (9)
C16	0.0343 (9)	0.0305 (10)	0.0268 (8)	−0.0077 (8)	−0.0078 (7)	−0.0089 (8)

Geometric parameters (Å, º)

Br1—C4	1.9019 (16)	C6—C9	1.498 (3)
S1—O2	1.4396 (12)	C8—C10	1.480 (2)
S1—O3	1.4403 (12)	C9—H9A	0.9800
S1—C1	1.7354 (18)	C9—H9B	0.9800
S1—C11	1.7612 (17)	C9—H9C	0.9800
F1—C14	1.354 (2)	C10—H10A	0.9800
O1—C8	1.368 (2)	C10—H10B	0.9800
O1—C7	1.377 (2)	C10—H10C	0.9800

C1—C8	1.362 (2)	C11—C12	1.387 (2)
C1—C2	1.448 (2)	C11—C16	1.391 (2)
C2—C7	1.395 (2)	C12—C13	1.379 (3)
C2—C3	1.398 (2)	C12—H12	0.9500
C3—C4	1.382 (3)	C13—C14	1.371 (3)
C3—H3	0.9500	C13—H13	0.9500
C4—C5	1.392 (3)	C14—C15	1.371 (3)
C5—C6	1.389 (2)	C15—C16	1.385 (3)
C5—H5	0.9500	C15—H15	0.9500
C6—C7	1.383 (2)	C16—H16	0.9500

O2—S1—O3	119.77 (8)	C6—C9—H9A	109.5
O2—S1—C1	106.57 (8)	C6—C9—H9B	109.5
O3—S1—C1	108.82 (8)	H9A—C9—H9B	109.5
O2—S1—C11	107.04 (8)	C6—C9—H9C	109.5
O3—S1—C11	107.58 (8)	H9A—C9—H9C	109.5
C1—S1—C11	106.33 (8)	H9B—C9—H9C	109.5
C8—O1—C7	107.04 (13)	C8—C10—H10A	109.5
C8—C1—C2	107.36 (15)	C8—C10—H10B	109.5
C8—C1—S1	126.67 (13)	H10A—C10—H10B	109.5
C2—C1—S1	125.96 (13)	C8—C10—H10C	109.5
C7—C2—C3	119.41 (15)	H10A—C10—H10C	109.5
C7—C2—C1	104.63 (14)	H10B—C10—H10C	109.5
C3—C2—C1	135.96 (16)	C12—C11—C16	121.12 (16)
C4—C3—C2	115.86 (16)	C12—C11—S1	119.34 (13)
C4—C3—H3	122.1	C16—C11—S1	119.49 (13)
C2—C3—H3	122.1	C13—C12—C11	119.55 (17)
C3—C4—C5	123.81 (16)	C13—C12—H12	120.2
C3—C4—Br1	118.15 (14)	C11—C12—H12	120.2
C5—C4—Br1	118.04 (13)	C14—C13—C12	118.25 (18)
C6—C5—C4	121.09 (17)	C14—C13—H13	120.9
C6—C5—H5	119.5	C12—C13—H13	120.9
C4—C5—H5	119.5	F1—C14—C13	118.26 (19)
C7—C6—C5	114.69 (16)	F1—C14—C15	118.05 (19)
C7—C6—C9	122.24 (16)	C13—C14—C15	123.68 (18)
C5—C6—C9	123.06 (17)	C14—C15—C16	118.15 (18)
O1—C7—C6	124.43 (16)	C14—C15—H15	120.9
O1—C7—C2	110.43 (14)	C16—C15—H15	120.9
C6—C7—C2	125.14 (15)	C15—C16—C11	119.24 (17)
C1—C8—O1	110.54 (15)	C15—C16—H16	120.4
C1—C8—C10	134.51 (18)	C11—C16—H16	120.4
O1—C8—C10	114.95 (15)		

O2—S1—C1—C8	157.48 (15)	C1—C2—C7—O1	−0.36 (18)
O3—S1—C1—C8	27.03 (18)	C3—C2—C7—C6	−1.2 (3)
C11—S1—C1—C8	−88.59 (17)	C1—C2—C7—C6	178.73 (16)
O2—S1—C1—C2	−22.76 (17)	C2—C1—C8—O1	0.24 (19)
O3—S1—C1—C2	−153.21 (14)	S1—C1—C8—O1	−179.96 (12)

C11—S1—C1—C2	91.18 (15)	C2—C1—C8—C10	−179.02 (19)
C8—C1—C2—C7	0.08 (18)	S1—C1—C8—C10	0.8 (3)
S1—C1—C2—C7	−179.73 (13)	C7—O1—C8—C1	−0.46 (19)
C8—C1—C2—C3	179.94 (19)	C7—O1—C8—C10	178.96 (15)
S1—C1—C2—C3	0.1 (3)	O2—S1—C11—C12	−147.79 (15)
C7—C2—C3—C4	0.8 (2)	O3—S1—C11—C12	−17.86 (17)
C1—C2—C3—C4	−179.07 (18)	C1—S1—C11—C12	98.59 (16)
C2—C3—C4—C5	0.1 (3)	O2—S1—C11—C16	29.83 (17)
C2—C3—C4—Br1	179.36 (12)	O3—S1—C11—C16	159.77 (14)
C3—C4—C5—C6	−0.8 (3)	C1—S1—C11—C16	−83.78 (16)
Br1—C4—C5—C6	179.98 (13)	C16—C11—C12—C13	−0.6 (3)
C4—C5—C6—C7	0.5 (2)	S1—C11—C12—C13	176.99 (16)
C4—C5—C6—C9	−179.85 (16)	C11—C12—C13—C14	0.8 (3)
C8—O1—C7—C6	−178.59 (16)	C12—C13—C14—F1	179.41 (19)
C8—O1—C7—C2	0.51 (18)	C12—C13—C14—C15	0.1 (3)
C5—C6—C7—O1	179.48 (15)	F1—C14—C15—C16	179.55 (18)
C9—C6—C7—O1	−0.2 (3)	C13—C14—C15—C16	−1.1 (3)
C5—C6—C7—C2	0.5 (2)	C14—C15—C16—C11	1.3 (3)
C9—C6—C7—C2	−179.19 (16)	C12—C11—C16—C15	−0.5 (3)
C3—C2—C7—O1	179.75 (14)	S1—C11—C16—C15	−178.04 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O2^{i}	0.95	2.53	3.408 (2)	154
C9—H9*A*···O2^{ii}	0.98	2.50	3.304 (2)	139

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