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

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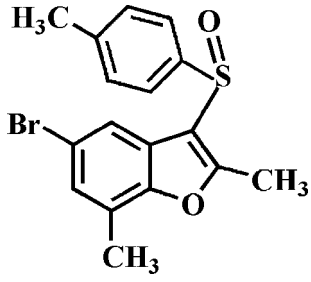
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.005 Å; R factor = 0.045; wR factor = 0.089; data-to-parameter ratio = 17.3.

In the title compound, $C_{17}H_{15}BrO_2S$, the 4-methylbenzene ring makes a dihedral angle of 89.01 (7)° with the mean plane [r.m.s. deviation = 0.013 (2) Å] of the benzofuran fragment. In the crystal, molecules are linked into supramolecular layers that stack along [001] by weak C—H···O, C—H···π and C—S···π [3.364 (2) Å] interactions.

Related literature

For background information and the crystal structures of related compounds, see: Choi *et al.* (2011*a*,*b*).



Experimental

Crystal data

$C_{17}H_{15}BrO_2S$
M_r = 363.26
Triclinic, $P\overline{1}$
a = 6.1794 (6) Å
b = 10.057 (1) Å
c = 12.5793 (12) Å
α = 84.072 (6)°
β = 79.738 (6)°
γ = 85.471 (6)°
V = 763.67 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.83 mm^{-1}
T = 173 K
0.22 × 0.13 × 0.12 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.640, T_{max} = 0.746
13108 measured reflections
3331 independent reflections
2180 reflections with $I > 2\sigma(I)$
R_{int} = 0.075

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.089
S = 1.04
3331 reflections
193 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

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

Cg is the centroid of the C11–C16 4-methylphenyl ring.

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···O2^{i}	0.95	2.58	3.338 (4)	137
C17—H17*C*···*Cg*^{ii}	0.98	2.77	3.739 (4)	169

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

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK5212).

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Choi, H. D., Seo, P. J., Son, B. W. & Lee, U. (2011*b*). *Acta Cryst.* E**67**, o1039.
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supporting information

Acta Cryst. (2013). E**69**, o720 [https://doi.org/10.1107/S1600536813009756]

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

Hong Dae Choi, Pil Ja Seo and Uk Lee

S1. Comment

As a part of our continuing study of 5-bromo-2,7-dimethyl-1-benzofuran derivatives containing 4-fluorophenylsulfinyl (Choi *et al.*, 2011*a*) and 4-cyclohexylsulfinyl (Choi *et al.*, 2011*b*) substituents in the 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 0.013 (2) Å from the least-squares plane defined by the nine constituent atoms. The dihedral angle between the 4-methylbenzene ring and the mean plane of the benzofuran ring is 89.01 (7)°. In the crystal structure (Fig. 2), molecules are connected by weak C–H···O and C–H···π interactions (Table 1, Cg is the centroid of the C11-C16 4-methylphenyl ring), and by intermolecular C–S···π interactions between the sulfur atom and the 4-methylphenyl ring of an adjacent molecule, with a S1···Cg^{iii} being 3.364 (2) Å.

S2. Experimental

3-Chloroperoxybenzoic acid (77%, 224 mg, 1.0 mmol) was added in small portions to a stirred solution of 5-bromo-2,7-dimethyl-3-(4-methylphenylsulfanyl)-1-benzofuran (302 mg, 0.9 mmol) in dichloromethane (30 mL) at 273 K. After being stirred at room temperature for 4 h, 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, 1:1 *v*/*v*) to afford the title compound as a colourless solid [yield 79%, *M*.pt: 406-407 K; R_f = 0.63 (hexane-ethyl acetate, 1:1 *v*/v)]. Single crystals suitable for X-ray diffraction were prepared by slow evaporation of its acetone solution held 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.

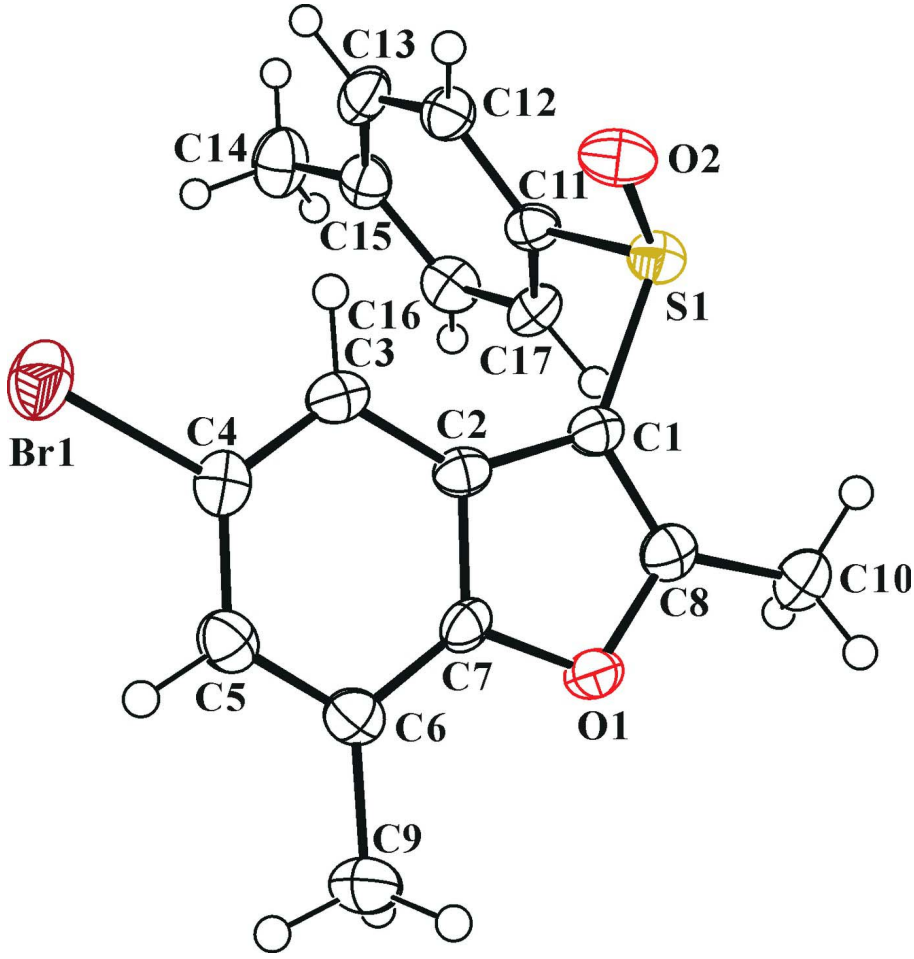

Figure 1

The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as small spheres of arbitrary radius.

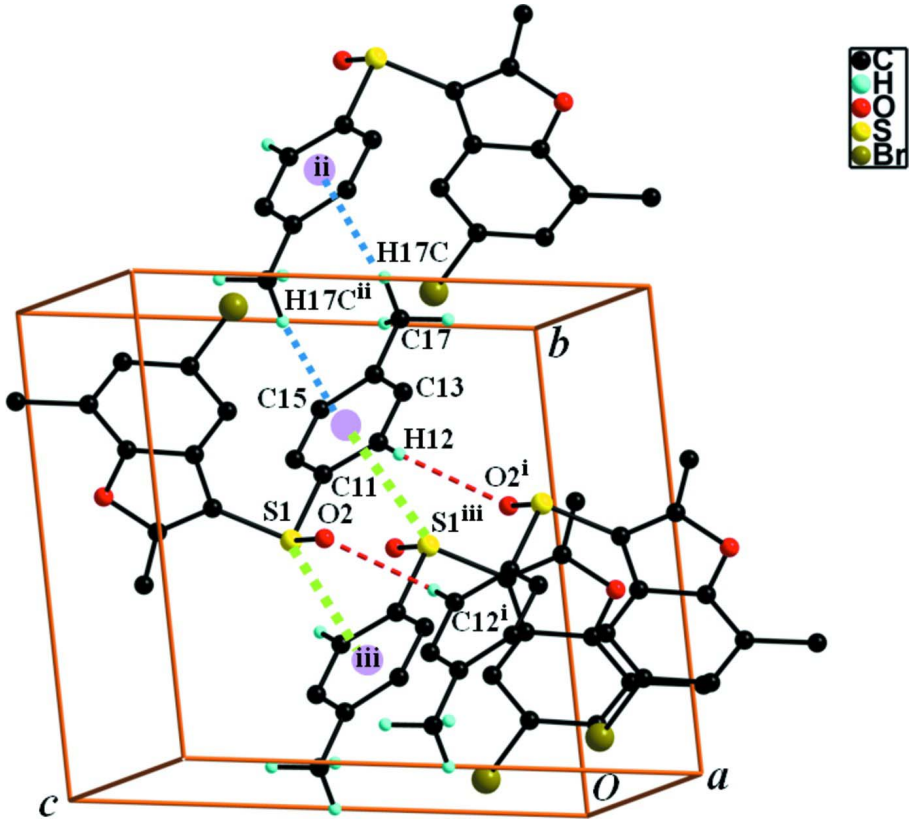

Figure 2

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

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

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Hall symbol: -P 1
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β = 79.738 (6)°
γ = 85.471 (6)°
V = 763.67 (13) Å^3
Z = 2
F(000) = 368
D_x = 1.580 Mg m^{-3}
Melting point = 406–407 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3261 reflections
θ = 2.4–26.4°
μ = 2.83 mm^{-1}
T = 173 K
Block, colourless
0.22 × 0.13 × 0.12 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} = 0.640, T_{max} = 0.746
13108 measured reflections
3331 independent reflections
2180 reflections with $I > 2\sigma(I)$

R_{int} = 0.075
θ_{max} = 27.0°, θ_{min} = 2.0°
h = −7→7
k = −12→12
l = −16→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.089
S = 1.04
3331 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
$w = 1/[\sigma^2(F_o^2) + (0.0171P)^2 + 0.341P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 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}
Br1	1.08078 (6)	0.92372 (4)	0.82890 (3)	0.04049 (15)
S1	0.60771 (14)	0.49101 (9)	0.65693 (6)	0.0281 (2)
O1	0.2942 (3)	0.5989 (2)	0.93938 (16)	0.0285 (6)
O2	0.8519 (3)	0.4747 (2)	0.64788 (17)	0.0355 (6)
C1	0.5062 (5)	0.5624 (3)	0.7794 (2)	0.0243 (8)
C2	0.6048 (5)	0.6623 (3)	0.8261 (2)	0.0237 (8)
C3	0.7900 (5)	0.7364 (3)	0.7958 (3)	0.0273 (8)
H3	0.8874	0.7271	0.7291	0.033*
C4	0.8251 (5)	0.8235 (3)	0.8669 (3)	0.0284 (8)
C5	0.6844 (5)	0.8414 (3)	0.9642 (3)	0.0303 (9)
H5	0.7164	0.9043	1.0098	0.036*
C6	0.4982 (5)	0.7692 (3)	0.9961 (2)	0.0273 (8)
C7	0.4681 (5)	0.6809 (3)	0.9246 (3)	0.0241 (8)
C8	0.3243 (5)	0.5271 (3)	0.8496 (3)	0.0271 (8)
C9	0.3398 (6)	0.7855 (4)	1.1002 (3)	0.0399 (10)
H9A	0.1935	0.8152	1.0838	0.060*
H9B	0.3911	0.8522	1.1399	0.060*
H9C	0.3315	0.6995	1.1447	0.060*
C10	0.1534 (6)	0.4332 (3)	0.8474 (3)	0.0335 (9)
H10A	0.0188	0.4838	0.8317	0.050*
H10B	0.1218	0.3813	0.9181	0.050*
H10C	0.2062	0.3723	0.7911	0.050*

C11	0.5490 (5)	0.6350 (3)	0.5679 (2)	0.0247 (8)
C12	0.7171 (5)	0.6888 (3)	0.4924 (3)	0.0290 (8)
H12	0.8642	0.6515	0.4883	0.035*
C13	0.6670 (6)	0.7983 (4)	0.4229 (3)	0.0340 (9)
H13	0.7823	0.8369	0.3716	0.041*
C14	0.4543 (6)	0.8527 (3)	0.4259 (3)	0.0304 (8)
C15	0.2879 (6)	0.7931 (4)	0.4998 (3)	0.0326 (9)
H15	0.1399	0.8279	0.5020	0.039*
C16	0.3330 (5)	0.6843 (3)	0.5699 (3)	0.0303 (8)
H16	0.2170	0.6435	0.6192	0.036*
C17	0.4004 (6)	0.9735 (4)	0.3523 (3)	0.0432 (10)
H17A	0.2433	0.9781	0.3477	0.065*
H17B	0.4880	0.9666	0.2798	0.065*
H17C	0.4348	1.0546	0.3814	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0302 (2)	0.0392 (3)	0.0535 (3)	−0.00854 (17)	−0.00904 (17)	−0.00239 (19)
S1	0.0288 (5)	0.0298 (6)	0.0253 (5)	−0.0003 (4)	−0.0035 (4)	−0.0036 (4)
O1	0.0286 (14)	0.0304 (15)	0.0237 (13)	−0.0015 (11)	0.0017 (10)	−0.0003 (11)
O2	0.0250 (13)	0.0460 (17)	0.0343 (14)	0.0097 (11)	−0.0052 (10)	−0.0069 (12)
C1	0.0237 (19)	0.026 (2)	0.0219 (18)	0.0001 (15)	−0.0024 (14)	0.0001 (16)
C2	0.0204 (18)	0.027 (2)	0.0226 (18)	0.0015 (15)	−0.0032 (14)	−0.0007 (16)
C3	0.0243 (19)	0.031 (2)	0.0237 (18)	0.0013 (16)	−0.0006 (14)	0.0009 (17)
C4	0.026 (2)	0.026 (2)	0.034 (2)	−0.0021 (16)	−0.0092 (16)	0.0000 (17)
C5	0.036 (2)	0.028 (2)	0.028 (2)	0.0004 (17)	−0.0082 (16)	−0.0043 (17)
C6	0.035 (2)	0.026 (2)	0.0211 (18)	0.0019 (17)	−0.0076 (15)	0.0001 (16)
C7	0.0242 (19)	0.021 (2)	0.0242 (19)	−0.0022 (16)	−0.0014 (14)	0.0050 (16)
C8	0.028 (2)	0.026 (2)	0.0256 (19)	0.0013 (16)	−0.0066 (15)	0.0030 (17)
C9	0.048 (2)	0.043 (3)	0.027 (2)	−0.0022 (19)	0.0015 (17)	−0.0063 (18)
C10	0.032 (2)	0.033 (2)	0.035 (2)	−0.0082 (17)	−0.0044 (15)	0.0015 (18)
C11	0.0236 (19)	0.031 (2)	0.0201 (17)	−0.0027 (16)	−0.0042 (14)	−0.0040 (16)
C12	0.0238 (19)	0.033 (2)	0.031 (2)	−0.0040 (16)	−0.0046 (15)	−0.0064 (17)
C13	0.031 (2)	0.037 (2)	0.034 (2)	−0.0136 (18)	−0.0034 (16)	0.0002 (19)
C14	0.036 (2)	0.032 (2)	0.0258 (19)	−0.0061 (17)	−0.0085 (16)	−0.0029 (17)
C15	0.028 (2)	0.036 (2)	0.036 (2)	0.0038 (17)	−0.0113 (16)	−0.0051 (18)
C16	0.025 (2)	0.035 (2)	0.0296 (19)	−0.0075 (16)	−0.0008 (15)	0.0011 (17)
C17	0.052 (3)	0.039 (3)	0.041 (2)	−0.0099 (19)	−0.0174 (19)	0.0050 (19)

Geometric parameters (Å, º)

Br1—C4	1.904 (3)	C9—H9B	0.9800
S1—O2	1.490 (2)	C9—H9C	0.9800
S1—C1	1.760 (3)	C10—H10A	0.9800
S1—C11	1.792 (3)	C10—H10B	0.9800
O1—C8	1.380 (4)	C10—H10C	0.9800
O1—C7	1.381 (4)	C11—C12	1.380 (4)

C1—C8	1.347 (4)	C11—C16	1.383 (4)
C1—C2	1.439 (4)	C12—C13	1.385 (4)
C2—C3	1.390 (4)	C12—H12	0.9500
C2—C7	1.390 (4)	C13—C14	1.379 (4)
C3—C4	1.369 (4)	C13—H13	0.9500
C3—H3	0.9500	C14—C15	1.387 (5)
C4—C5	1.389 (5)	C14—C17	1.503 (5)
C5—C6	1.385 (4)	C15—C16	1.375 (4)
C5—H5	0.9500	C15—H15	0.9500
C6—C7	1.372 (4)	C16—H16	0.9500
C6—C9	1.504 (4)	C17—H17A	0.9800
C8—C10	1.477 (5)	C17—H17B	0.9800
C9—H9A	0.9800	C17—H17C	0.9800

O2—S1—C1	106.70 (14)	H9A—C9—H9C	109.5
O2—S1—C11	106.82 (14)	H9B—C9—H9C	109.5
C1—S1—C11	96.87 (15)	C8—C10—H10A	109.5
C8—O1—C7	106.4 (2)	C8—C10—H10B	109.5
C8—C1—C2	107.4 (3)	H10A—C10—H10B	109.5
C8—C1—S1	125.2 (3)	C8—C10—H10C	109.5
C2—C1—S1	127.3 (2)	H10A—C10—H10C	109.5
C3—C2—C7	118.9 (3)	H10B—C10—H10C	109.5
C3—C2—C1	135.8 (3)	C12—C11—C16	120.7 (3)
C7—C2—C1	105.3 (3)	C12—C11—S1	119.6 (2)
C4—C3—C2	116.7 (3)	C16—C11—S1	119.4 (2)
C4—C3—H3	121.7	C11—C12—C13	118.6 (3)
C2—C3—H3	121.7	C11—C12—H12	120.7
C3—C4—C5	123.3 (3)	C13—C12—H12	120.7
C3—C4—Br1	117.6 (3)	C14—C13—C12	121.8 (3)
C5—C4—Br1	119.1 (3)	C14—C13—H13	119.1
C6—C5—C4	121.2 (3)	C12—C13—H13	119.1
C6—C5—H5	119.4	C13—C14—C15	118.2 (3)
C4—C5—H5	119.4	C13—C14—C17	121.9 (3)
C7—C6—C5	114.6 (3)	C15—C14—C17	120.0 (3)
C7—C6—C9	122.1 (3)	C16—C15—C14	121.2 (3)
C5—C6—C9	123.3 (3)	C16—C15—H15	119.4
C6—C7—O1	124.7 (3)	C14—C15—H15	119.4
C6—C7—C2	125.3 (3)	C15—C16—C11	119.3 (3)
O1—C7—C2	110.0 (3)	C15—C16—H16	120.3
C1—C8—O1	110.8 (3)	C11—C16—H16	120.3
C1—C8—C10	133.4 (3)	C14—C17—H17A	109.5
O1—C8—C10	115.8 (3)	C14—C17—H17B	109.5
C6—C9—H9A	109.5	H17A—C17—H17B	109.5
C6—C9—H9B	109.5	C14—C17—H17C	109.5
H9A—C9—H9B	109.5	H17A—C17—H17C	109.5
C6—C9—H9C	109.5	H17B—C17—H17C	109.5

O2—S1—C1—C8	−139.0 (3)	C1—C2—C7—C6	−179.1 (3)

C11—S1—C1—C8	111.1 (3)	C3—C2—C7—O1	179.8 (3)
O2—S1—C1—C2	37.5 (3)	C1—C2—C7—O1	−0.1 (3)
C11—S1—C1—C2	−72.4 (3)	C2—C1—C8—O1	0.9 (3)
C8—C1—C2—C3	179.7 (3)	S1—C1—C8—O1	177.9 (2)
S1—C1—C2—C3	2.7 (5)	C2—C1—C8—C10	178.6 (3)
C8—C1—C2—C7	−0.4 (3)	S1—C1—C8—C10	−4.3 (5)
S1—C1—C2—C7	−177.4 (2)	C7—O1—C8—C1	−0.9 (3)
C7—C2—C3—C4	0.3 (4)	C7—O1—C8—C10	−179.1 (2)
C1—C2—C3—C4	−179.8 (3)	O2—S1—C11—C12	13.2 (3)
C2—C3—C4—C5	−1.1 (5)	C1—S1—C11—C12	123.1 (3)
C2—C3—C4—Br1	178.7 (2)	O2—S1—C11—C16	−171.8 (3)
C3—C4—C5—C6	0.9 (5)	C1—S1—C11—C16	−62.0 (3)
Br1—C4—C5—C6	−179.0 (2)	C16—C11—C12—C13	3.8 (5)
C4—C5—C6—C7	0.2 (4)	S1—C11—C12—C13	178.6 (2)
C4—C5—C6—C9	−179.6 (3)	C11—C12—C13—C14	−1.2 (5)
C5—C6—C7—O1	−179.9 (3)	C12—C13—C14—C15	−1.3 (5)
C9—C6—C7—O1	−0.1 (5)	C12—C13—C14—C17	178.5 (3)
C5—C6—C7—C2	−1.1 (5)	C13—C14—C15—C16	1.4 (5)
C9—C6—C7—C2	178.7 (3)	C17—C14—C15—C16	−178.5 (3)
C8—O1—C7—C6	179.6 (3)	C14—C15—C16—C11	1.1 (5)
C8—O1—C7—C2	0.6 (3)	C12—C11—C16—C15	−3.7 (5)
C3—C2—C7—C6	0.8 (5)	S1—C11—C16—C15	−178.6 (3)

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C11–C16 4-methylphenyl ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···O2^{i}	0.95	2.58	3.338 (4)	137
C17—H17*C*···*Cg*^{ii}	0.98	2.77	3.739 (4)	169

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