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5-Bromo-3-ethylsulfinyl-2-(4-methylphenyl)-1-benzofuran

Hong Dae Choi,[a] Pil Ja Seo[a] and Uk Lee[b]*

[a]Department of Chemistry, Dongeui University, San 24 Kaya-dong, Busanjin-gu, Busan 614-714, Republic of Korea, and [b]Department of Chemistry, Pukyong National University, 599-1 Daeyeon 3-dong, Nam-gu, Busan 608-737, Republic of Korea
Correspondence e-mail: uklee@pknu.ac.kr

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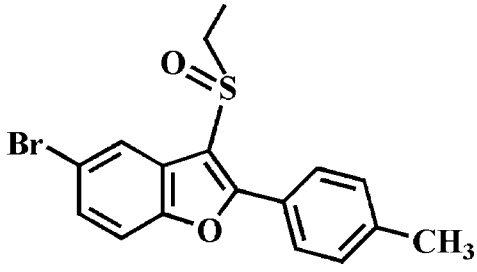
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.005 Å; R factor = 0.052; wR factor = 0.139; data-to-parameter ratio = 20.1.

In the title compound, $C_{17}H_{15}BrO_2S$, the dihedral angle between the plane of the benzofuran ring system [r.m.s. deviation = 0.004 (3) Å] and that of the 4-methylphenyl ring is 0.9 (2)°. In the crystal, molecules are linked by C—H···O, C—H···π and Br···π [3.636 (2) Å] interactions, and by π–π interactions between the 4-methylphenyl and furan rings of neighbouring molecules [centroid–centroid distance = 3.650 (2) Å], forming a three-dimensional network.

Related literature

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



Experimental

Crystal data

$C_{17}H_{15}BrO_2S$
M_r = 363.26
Monoclinic, $P2_1/n$
a = 5.0202 (3) Å
b = 25.0471 (12) Å
c = 12.2430 (6) Å
β = 100.340 (2)°
V = 1514.45 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.85 mm^{-1}
T = 173 K
0.60 × 0.14 × 0.12 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.541, T_{max} = 0.746
26373 measured reflections
3868 independent reflections
3104 reflections with $I > 2\sigma(I)$
R_{int} = 0.058

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.139
S = 1.06
3868 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.97 e Å^{-3}
$\Delta\rho_{min}$ = −0.83 e Å^{-3}

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

*Cg*1 is the centroid of the C9–C14 4-methylphenyl ring.

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13···O2^{i}	0.95	2.58	3.507 (4)	164
C16—H16*B*···O2^{ii}	0.99	2.26	3.110 (5)	144
C15—H15*C*···*Cg*1^{ii}	0.99	2.78	3.607 (3)	143

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

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: *SHELXS97*.

Supporting information for this paper is available from the IUCr electronic archives (Reference: FF2130).

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supporting information

Acta Cryst. (2014). E**70**, o808 [https://doi.org/10.1107/S1600536814014470]

5-Bromo-3-ethylsulfinyl-2-(4-methylphenyl)-1-benzofuran

Hong Dae Choi, Pil Ja Seo and Uk Lee

S1. Comment

As a part of our ongoing project of 2-aryl-5-bromo-1-benzofuran derivatives containing [3-ethylsulfinyl-2-(4-fluorophenyl)] (Choi *et al.*, 2010) and [2-(4-methylphenyl)-3-methylsulfinyl] (Choi *et al.*, 2012) substituents. 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 0.004 (3) Å from the least-squares plane defined by the nine constituent atoms. The 4-methylphenyl ring is essentially planar, with a mean deviation of 0.002 (3) Å from the least-squares plane defined by the six constituent atoms. The dihedral angle formed by the benzofuran ring system and the 4-methylphenyl ring is 0.9 (2)°. In the crystal structure (Fig. 2), molecules are linked by C—H···O and C—H···π hydrogen bonds (Table 1, Cg1 is the centroid of the C9–C14 4-methylphenyl ring) and C4—Br1···π interactions between the bromine atom and the benzene ring of a neighbouring molecule with a Br1···Cg3^{iii} = 3.636 (2) Å (Cg3 is the centroid of the C2–C7 benzene ring). The crystal packing (Fig. 2) also exhibits π···π interactions between the 4-methylphenyl and furan rings of neighbouring molecules, with a Cg1···Cg2^{ii} distance of 3.650 (2) Å (Cg2 is the centroid of the C1/C2/C7/O1/C8 furan ring), forming a three-dimensional network.

S2. Experimental

3-Chloroperoxybenzoic acid (77%, 269 mg, 1.2 mmol) was added in small portions to a stirred solution of 5-bromo-3-ethylsulfanyl-2-(4-methylphenyl)-1-benzofuran (382 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, 1:1 *v*/*v*) to afford the title compound as a colorless solid [yield 71%, m.p. 404–405 K; R_f = 0.54 (hexane–ethyl acetate, 1:1 *v*/*v*)]. Single crystals suitable for X-ray diffraction were prepared by slow evaporation of a solution of the title compound in benzene 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, 0.99 Å for methylene and 0.98 Å for methyl H atoms, respectively. U_{iso} (H) = 1.2U_{eq} (C) for aryl and methylene, 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). The highest peak in the difference map is 0.91 Å from Br1 and the largest hole is 0.59 Å from Br1.

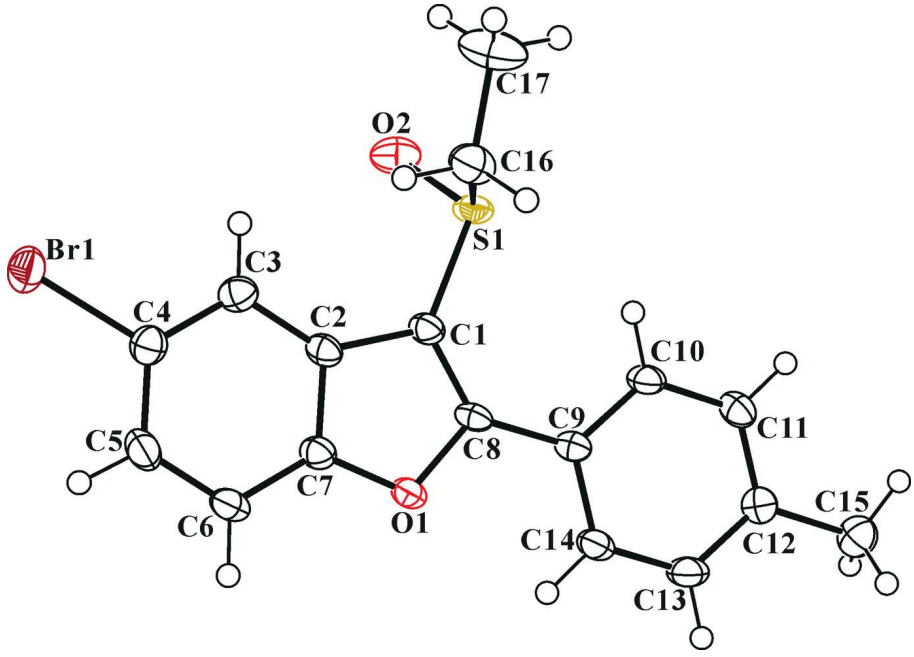

Figure 1

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.

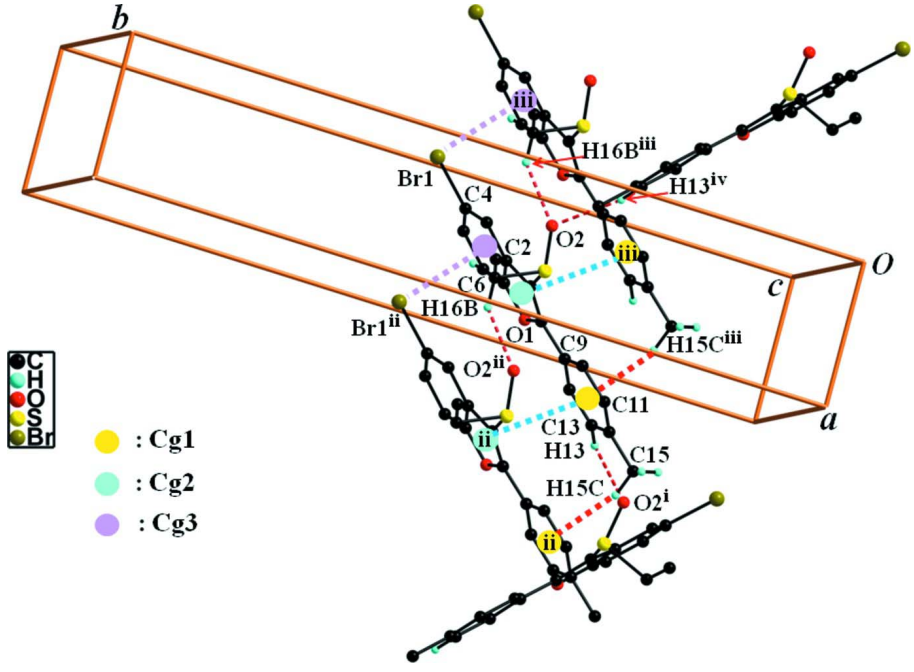

Figure 2

A view of the C—H···O, C—H···π, Br···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 + 3/2, - y + 1/2, z + 1/2; (ii) x + 1, y, z; (iii) x - 1, y, z; (iv) x - 3/2, - y + 1/2, z - 1/2.]

5-Bromo-3-ethylsulfinyl-2-(4-methylphenyl)-1-benzofuran

Crystal data

$C_{17}H_{15}BrO_2S$
$M_r = 363.26$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 5.0202$ (3) Å
$b = 25.0471$ (12) Å
$c = 12.2430$ (6) Å
$\beta = 100.340$ (2)°
$V = 1514.45$ (14) Å^3
$Z = 4$
$F(000) = 736$
$D_x = 1.593$ Mg m^{-3}
Melting point = 405–404 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7637 reflections
$\theta = 2.4$–$27.2°$
$\mu = 2.85$ mm^{-1}
$T = 173$ K
Block, colourless
0.60 × 0.14 × 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)
$T_{min} = 0.541$, $T_{max} = 0.746$
26373 measured reflections
3868 independent reflections
3104 reflections with $I > 2\sigma(I)$
$R_{int} = 0.058$
$\theta_{max} = 28.6°$, $\theta_{min} = 1.6°$
$h = -6 \rightarrow 6$
$k = -33 \rightarrow 32$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.139$
$S = 1.06$
3868 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
$w = 1/[\sigma^2(F_o^2) + (0.0648P)^2 + 3.2187P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 1.97$ e Å^{-3}
$\Delta\rho_{min} = -0.83$ 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	−0.00958 (8)	0.504511 (15)	0.82039 (3)	0.03501 (14)
S1	0.56805 (19)	0.35409 (3)	0.54542 (7)	0.0275 (2)
O1	0.8410 (5)	0.33583 (9)	0.86960 (18)	0.0242 (5)

O2	0.2710 (5)	0.36245 (11)	0.5189 (2)	0.0368 (6)
C1	0.6586 (7)	0.35476 (13)	0.6920 (3)	0.0229 (6)
C2	0.5277 (7)	0.38802 (13)	0.7630 (3)	0.0220 (6)
C3	0.3264 (7)	0.42681 (13)	0.7465 (3)	0.0258 (7)
H3	0.2391	0.4368	0.6740	0.031*
C4	0.2582 (7)	0.45033 (14)	0.8400 (3)	0.0266 (7)
C5	0.3796 (8)	0.43619 (15)	0.9476 (3)	0.0308 (8)
H5	0.3257	0.4532	1.0095	0.037*
C6	0.5790 (8)	0.39732 (15)	0.9641 (3)	0.0304 (8)
H6	0.6644	0.3869	1.0366	0.037*
C7	0.6481 (7)	0.37435 (13)	0.8707 (3)	0.0236 (7)
C8	0.8441 (7)	0.32375 (12)	0.7603 (2)	0.0208 (6)
C9	1.0400 (7)	0.28307 (13)	0.7429 (3)	0.0214 (6)
C10	1.0737 (7)	0.26723 (14)	0.6372 (3)	0.0253 (7)
H10	0.9656	0.2830	0.5738	0.030*
C11	1.2624 (7)	0.22877 (14)	0.6235 (3)	0.0272 (7)
H11	1.2815	0.2185	0.5505	0.033*
C12	1.4238 (7)	0.20489 (13)	0.7136 (3)	0.0250 (7)
C13	1.3907 (8)	0.22062 (15)	0.8188 (3)	0.0304 (8)
H13	1.4991	0.2045	0.8817	0.037*
C14	1.2043 (8)	0.25910 (14)	0.8346 (3)	0.0282 (7)
H14	1.1873	0.2694	0.9077	0.034*
C15	1.6299 (8)	0.16284 (15)	0.6982 (3)	0.0326 (8)
H15A	1.6346	0.1589	0.6189	0.049*
H15B	1.5796	0.1287	0.7280	0.049*
H15C	1.8091	0.1737	0.7376	0.049*
C16	0.7154 (8)	0.41740 (17)	0.5188 (3)	0.0343 (8)
H16A	0.6577	0.4453	0.5669	0.041*
H16B	0.9154	0.4149	0.5359	0.041*
C17	0.6247 (12)	0.4323 (2)	0.3983 (4)	0.0561 (13)
H17A	0.6821	0.4046	0.3510	0.084*
H17B	0.7062	0.4665	0.3835	0.084*
H17C	0.4270	0.4355	0.3822	0.084*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0368 (2)	0.0297 (2)	0.0398 (2)	0.00835 (15)	0.01023 (16)	−0.00114 (15)
S1	0.0371 (5)	0.0267 (4)	0.0158 (4)	0.0048 (3)	−0.0031 (3)	−0.0012 (3)
O1	0.0324 (13)	0.0245 (12)	0.0153 (10)	0.0040 (9)	0.0033 (9)	−0.0006 (9)
O2	0.0348 (15)	0.0414 (16)	0.0300 (13)	−0.0066 (12)	−0.0056 (11)	0.0023 (12)
C1	0.0293 (17)	0.0211 (15)	0.0174 (14)	0.0008 (12)	0.0019 (12)	−0.0020 (12)
C2	0.0274 (16)	0.0197 (15)	0.0181 (14)	−0.0028 (12)	0.0021 (12)	−0.0032 (12)
C3	0.0268 (16)	0.0233 (16)	0.0257 (16)	−0.0008 (13)	0.0005 (13)	−0.0001 (13)
C4	0.0255 (16)	0.0254 (16)	0.0289 (17)	−0.0001 (13)	0.0051 (13)	−0.0010 (13)
C5	0.041 (2)	0.0293 (18)	0.0246 (17)	0.0030 (15)	0.0126 (15)	−0.0033 (14)
C6	0.040 (2)	0.0336 (19)	0.0170 (15)	0.0051 (15)	0.0032 (14)	−0.0002 (13)
C7	0.0298 (17)	0.0207 (15)	0.0195 (15)	0.0009 (13)	0.0022 (13)	−0.0016 (12)

C8	0.0287 (16)	0.0192 (14)	0.0134 (13)	−0.0034 (12)	0.0004 (11)	−0.0011 (11)
C9	0.0266 (16)	0.0194 (14)	0.0172 (14)	−0.0023 (12)	0.0015 (12)	0.0001 (11)
C10	0.0318 (17)	0.0253 (16)	0.0178 (15)	0.0022 (13)	0.0021 (13)	0.0045 (12)
C11	0.0346 (18)	0.0261 (17)	0.0215 (16)	−0.0004 (14)	0.0069 (13)	−0.0033 (13)
C12	0.0262 (16)	0.0223 (16)	0.0265 (17)	−0.0013 (13)	0.0048 (13)	−0.0014 (13)
C13	0.0362 (19)	0.0324 (19)	0.0203 (16)	0.0053 (15)	−0.0014 (14)	0.0046 (14)
C14	0.0377 (19)	0.0297 (18)	0.0164 (15)	0.0064 (14)	0.0024 (13)	−0.0010 (13)
C15	0.0338 (19)	0.0272 (18)	0.037 (2)	0.0016 (15)	0.0054 (15)	−0.0017 (15)
C16	0.0335 (19)	0.042 (2)	0.0271 (18)	−0.0040 (16)	0.0052 (15)	0.0024 (16)
C17	0.079 (4)	0.056 (3)	0.032 (2)	−0.011 (3)	0.004 (2)	0.016 (2)

Geometric parameters (Å, º)

Br1—C4	1.895 (4)	C9—C14	1.403 (4)
S1—O2	1.483 (3)	C10—C11	1.382 (5)
S1—C1	1.770 (3)	C10—H10	0.9500
S1—C16	1.804 (4)	C11—C12	1.383 (5)
O1—C7	1.369 (4)	C11—H11	0.9500
O1—C8	1.375 (4)	C12—C13	1.384 (5)
C1—C8	1.375 (4)	C12—C15	1.512 (5)
C1—C2	1.445 (4)	C13—C14	1.381 (5)
C2—C3	1.390 (5)	C13—H13	0.9500
C2—C7	1.391 (4)	C14—H14	0.9500
C3—C4	1.383 (5)	C15—H15A	0.9800
C3—H3	0.9500	C15—H15B	0.9800
C4—C5	1.395 (5)	C15—H15C	0.9800
C5—C6	1.385 (5)	C16—C17	1.511 (6)
C5—H5	0.9500	C16—H16A	0.9900
C6—C7	1.379 (5)	C16—H16B	0.9900
C6—H6	0.9500	C17—H17A	0.9800
C8—C9	1.458 (5)	C17—H17B	0.9800
C9—C10	1.392 (5)	C17—H17C	0.9800
O2—S1—C1	106.61 (16)	C11—C10—H10	119.6
O2—S1—C16	105.37 (17)	C9—C10—H10	119.6
C1—S1—C16	97.81 (17)	C10—C11—C12	121.4 (3)
C7—O1—C8	107.2 (2)	C10—C11—H11	119.3
C8—C1—C2	106.9 (3)	C12—C11—H11	119.3
C8—C1—S1	129.3 (3)	C11—C12—C13	117.9 (3)
C2—C1—S1	123.6 (2)	C11—C12—C15	121.2 (3)
C3—C2—C7	119.4 (3)	C13—C12—C15	120.9 (3)
C3—C2—C1	135.5 (3)	C14—C13—C12	121.8 (3)
C7—C2—C1	105.1 (3)	C14—C13—H13	119.1
C4—C3—C2	117.3 (3)	C12—C13—H13	119.1
C4—C3—H3	121.3	C13—C14—C9	120.1 (3)
C2—C3—H3	121.3	C13—C14—H14	119.9
C3—C4—C5	122.8 (3)	C9—C14—H14	119.9
C3—C4—Br1	118.4 (3)	C12—C15—H15A	109.5

C5—C4—Br1	118.7 (3)	C12—C15—H15B	109.5
C6—C5—C4	119.8 (3)	H15A—C15—H15B	109.5
C6—C5—H5	120.1	C12—C15—H15C	109.5
C4—C5—H5	120.1	H15A—C15—H15C	109.5
C7—C6—C5	117.1 (3)	H15B—C15—H15C	109.5
C7—C6—H6	121.5	C17—C16—S1	109.4 (3)
C5—C6—H6	121.5	C17—C16—H16A	109.8
O1—C7—C6	125.8 (3)	S1—C16—H16A	109.8
O1—C7—C2	110.6 (3)	C17—C16—H16B	109.8
C6—C7—C2	123.5 (3)	S1—C16—H16B	109.8
C1—C8—O1	110.1 (3)	H16A—C16—H16B	108.2
C1—C8—C9	135.0 (3)	C16—C17—H17A	109.5
O1—C8—C9	114.9 (3)	C16—C17—H17B	109.5
C10—C9—C14	118.0 (3)	H17A—C17—H17B	109.5
C10—C9—C8	122.2 (3)	C16—C17—H17C	109.5
C14—C9—C8	119.8 (3)	H17A—C17—H17C	109.5
C11—C10—C9	120.8 (3)	H17B—C17—H17C	109.5

O2—S1—C1—C8	141.1 (3)	C2—C1—C8—O1	−0.8 (4)
C16—S1—C1—C8	−110.3 (3)	S1—C1—C8—O1	−177.1 (2)
O2—S1—C1—C2	−34.7 (3)	C2—C1—C8—C9	−179.4 (3)
C16—S1—C1—C2	74.0 (3)	S1—C1—C8—C9	4.3 (6)
C8—C1—C2—C3	−179.6 (4)	C7—O1—C8—C1	1.0 (4)
S1—C1—C2—C3	−3.0 (6)	C7—O1—C8—C9	179.9 (3)
C8—C1—C2—C7	0.3 (4)	C1—C8—C9—C10	0.2 (6)
S1—C1—C2—C7	176.9 (3)	O1—C8—C9—C10	−178.3 (3)
C7—C2—C3—C4	0.6 (5)	C1—C8—C9—C14	179.4 (4)
C1—C2—C3—C4	−179.5 (4)	O1—C8—C9—C14	0.8 (4)
C2—C3—C4—C5	−0.9 (5)	C14—C9—C10—C11	0.4 (5)
C2—C3—C4—Br1	178.6 (2)	C8—C9—C10—C11	179.6 (3)
C3—C4—C5—C6	0.5 (6)	C9—C10—C11—C12	−0.1 (5)
Br1—C4—C5—C6	−179.0 (3)	C10—C11—C12—C13	0.1 (5)
C4—C5—C6—C7	0.2 (6)	C10—C11—C12—C15	180.0 (3)
C8—O1—C7—C6	179.5 (3)	C11—C12—C13—C14	−0.4 (6)
C8—O1—C7—C2	−0.8 (4)	C15—C12—C13—C14	179.8 (4)
C5—C6—C7—O1	179.3 (3)	C12—C13—C14—C9	0.7 (6)
C5—C6—C7—C2	−0.4 (6)	C10—C9—C14—C13	−0.7 (5)
C3—C2—C7—O1	−179.8 (3)	C8—C9—C14—C13	−179.9 (3)
C1—C2—C7—O1	0.3 (4)	O2—S1—C16—C17	−60.2 (4)
C3—C2—C7—C6	0.0 (5)	C1—S1—C16—C17	−169.9 (3)
C1—C2—C7—C6	−179.9 (3)		

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the C9–C14 4-methylphenyl ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13···O2^{i}	0.95	2.58	3.507 (4)	164

C16—H16*B*···O2^{ii}	0.99	2.26	3.110 (5)	144
C15—H15*C*···*Cg*1^{ii}	0.99	2.78	3.607 (3)	143

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