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

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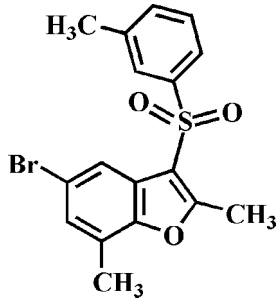
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.031; wR factor = 0.081; data-to-parameter ratio = 19.0.

In the title compound, $C_{17}H_{15}BrO_3S$, the dihedral angle between the mean planes of the benzofuran and 3-methylphenyl rings is 77.37 (5)°. In the crystal, molecules are linked *via* pairs of Br···O [Br···O = 3.335 (2) Å] contacts into inversion dimers. These dimers are further linked by C—H···O hydrogen bonds and π–π interactions between the benzene and furan rings of neighbouring molecules [centroid–centroid separation = 3.884 (3) Å] into supramolecular chains running along the *a*-axis direction.

Related literature

For background information and the crystal structures of related compounds, see: Choi *et al.* (2011, 2012, 2013). For a review of halogen bonding, see: Politzer *et al.* (2007).



Experimental

Crystal data

$C_{17}H_{15}BrO_3S$
M_r = 379.26
Monoclinic, $P2_1/c$
a = 8.8813 (3) Å
b = 6.5976 (2) Å
c = 26.5044 (8) Å
β = 97.635 (1)°
V = 1539.26 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.82 mm^{-1}
T = 296 K
0.50 × 0.46 × 0.15 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.379, T_{max} = 0.746
26392 measured reflections
3831 independent reflections
3188 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.081
S = 1.07
3831 reflections
202 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10B···O2^{i}	0.96	2.54	3.338 (3)	141
C17—H17C···O3^{ii}	0.96	2.41	3.357 (3)	170

Symmetry codes: (i) $x, y-1, z$; (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: *SHELXL97*.

Supporting information for this paper is available from the IUCr electronic archives (Reference: BH2497).

References

Brandenburg, K. (1998). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2009). *APEX2*, *SADABS* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Choi, H. D., Seo, P. J. & Lee, U. (2012). *Acta Cryst.* E**68**, o3208.
Choi, H. D., Seo, P. J. & Lee, U. (2013). *Acta Cryst.* E**69**, o720.
Choi, H. D., Seo, P. J., Son, B. W. & Lee, U. (2011). *Acta Cryst.* E**67**, o1279.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Politzer, P., Lane, P., Concha, M. C., Ma, Y. & Murray, J. S. (2007). *J. Mol. Model.* **13**, 305–311.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2014). E**70**, o567 [doi:10.1107/S1600536814008149]

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

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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-bromo-2,7-dimethyl-3-(3-methylphenylsulfanyl)-1-benzofuran (312 mg, 0.9 mmol) in dichloromethane (40 mL) at 273 K. After being stirred at room temperature for 8 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, 4:1 *v*/*v*) to afford the title compound as a colorless solid [yield 68%, m.p. 438–439 K; R_f = 0.51 (hexane–ethyl acetate, 4:1 *v*/*v*)]. 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.

S1.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. 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-bromo-2,7-dimethyl-1-benzofuran derivatives containing cyclohexylsulfonyl (Choi *et al.*, 2011), 4-fluorophenylsulfonyl (Choi *et al.*, 2012) and 4-methylphenylsulfinyl (Choi *et al.*, 2013) 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 0.010 (2) Å from the least-squares plane defined by the nine constituent atoms. The 3-methylphenyl ring is essentially planar, with a mean deviation of 0.004 (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 77.37 (5)°. In the crystal structure (Fig. 2), molecules are linked by pairs of Br···O halogen-bondings (Politzer *et al.* 2007) between the bromine and the O atom of the O═S═O unit [Br1···O2^{iii} = 3.335 (2) Å, C4—Br1···O2^{iii} = 168.67 (6)°, symmetry code: (iii) -*x*+1, -*y*+2, -*z*+1]. These dimers are further linked by C—H···O hydrogen bonds (Table 1), and by π···π interactions between the benzene and furan rings of neighbouring molecules, with a Cg1···Cg2^{iv} distance of 3.884 (3) Å and an interplanar distance of 3.440 (3) Å resulting in a slippage of 1.804 (3) Å (Cg1 and Cg2 are the centroids of the C2–C7 benzene ring and the C1/C2/C7/O1/C8 furan ring, respectively), forming supramolecular chains running along the *a*–axis direction.

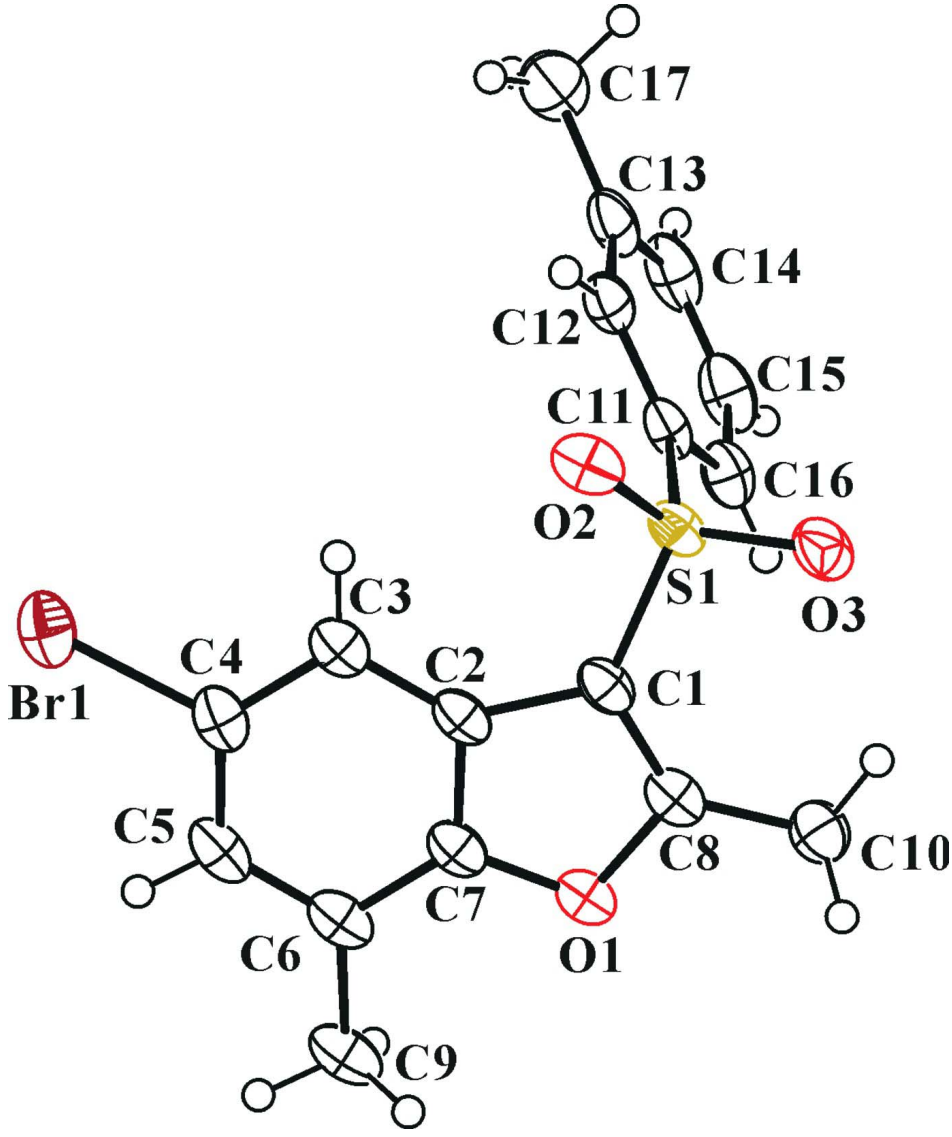

Figure 1

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.

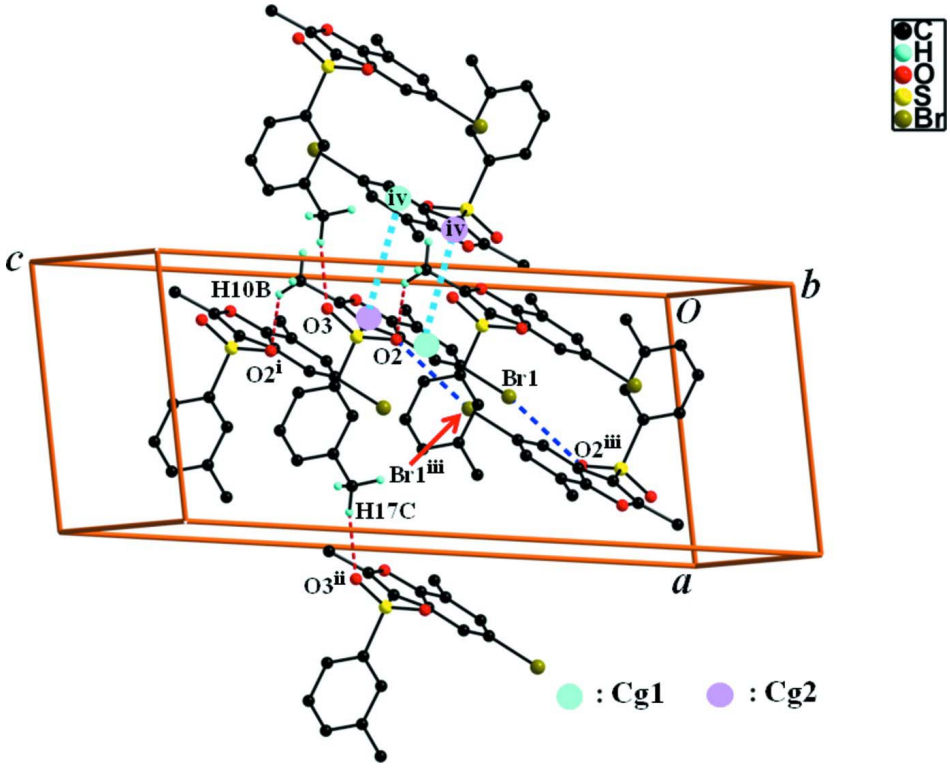

Figure 2

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

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Crystal data

$C_{17}H_{15}BrO_3S$
M_r = 379.26
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 8.8813 (3) Å
b = 6.5976 (2) Å
c = 26.5044 (8) Å
β = 97.635 (1)°
V = 1539.26 (8) Å^3
Z = 4
$F(000)$ = 768
D_x = 1.637 Mg m^{-3}
Melting point = 439–438 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9774 reflections
θ = 2.3–28.2°
μ = 2.82 mm^{-1}
T = 296 K
Block, colourless
0.50 × 0.46 × 0.15 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} = 0.379, T_{max} = 0.746
26392 measured reflections
3831 independent reflections
3188 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 28.3°, θ_{min} = 1.6°
h = −11→11
k = −8→8
l = −35→35

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.081$
$S = 1.07$
3831 reflections
202 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
$w = 1/[\sigma^2(F_o^2) + (0.0364P)^2 + 0.7777P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.56$ e Å^{-3}
$\Delta\rho_{min} = -0.45$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.46068 (3)	0.80654 (4)	0.428686 (8)	0.04118 (9)
S1	0.26202 (5)	0.72154 (8)	0.641971 (18)	0.02783 (11)
O1	0.09711 (16)	0.2852 (2)	0.55049 (5)	0.0314 (3)
O2	0.27849 (16)	0.9169 (2)	0.61959 (5)	0.0352 (3)
O3	0.16243 (16)	0.7009 (3)	0.68033 (6)	0.0380 (4)
C1	0.2041 (2)	0.5514 (3)	0.59334 (7)	0.0284 (4)
C2	0.2454 (2)	0.5613 (3)	0.54261 (7)	0.0277 (4)
C3	0.3313 (2)	0.6908 (3)	0.51640 (7)	0.0305 (4)
H3	0.3777	0.8062	0.5314	0.037*
C4	0.3435 (2)	0.6375 (4)	0.46673 (7)	0.0318 (4)
C5	0.2756 (2)	0.4660 (4)	0.44286 (7)	0.0347 (5)
H5	0.2888	0.4380	0.4093	0.042*
C6	0.1884 (2)	0.3360 (3)	0.46849 (8)	0.0325 (4)
C7	0.1763 (2)	0.3932 (3)	0.51806 (7)	0.0291 (4)
C8	0.1157 (2)	0.3850 (3)	0.59611 (7)	0.0302 (4)
C9	0.1153 (3)	0.1474 (4)	0.44553 (8)	0.0415 (5)
H9A	0.1709	0.0312	0.4596	0.062*
H9B	0.1157	0.1499	0.4093	0.062*
H9C	0.0125	0.1399	0.4529	0.062*
C10	0.0370 (3)	0.2911 (3)	0.63612 (9)	0.0368 (5)
H10A	0.0463	0.3777	0.6655	0.055*
H10B	0.0821	0.1619	0.6454	0.055*
H10C	−0.0685	0.2730	0.6235	0.055*
C11	0.4444 (2)	0.6401 (3)	0.66941 (7)	0.0283 (4)
C12	0.5609 (2)	0.7803 (3)	0.67428 (7)	0.0314 (4)
H12	0.5452	0.9090	0.6603	0.038*
C13	0.7026 (2)	0.7273 (4)	0.70039 (8)	0.0389 (5)
C14	0.7213 (3)	0.5329 (4)	0.71966 (8)	0.0461 (6)
H14	0.8152	0.4950	0.7369	0.055*
C15	0.6045 (3)	0.3937 (4)	0.71410 (8)	0.0444 (6)
H15	0.6208	0.2638	0.7274	0.053*
C16	0.4631 (2)	0.4458 (4)	0.68888 (8)	0.0367 (5)
H16	0.3834	0.3533	0.6852	0.044*
C17	0.8290 (2)	0.8819 (5)	0.70728 (10)	0.0535 (7)

H17A	0.8362	0.9388	0.7409	0.080*
H17B	0.8078	0.9877	0.6825	0.080*
H17C	0.9233	0.8176	0.7029	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.04301 (14)	0.04977 (17)	0.03230 (13)	0.01099 (10)	0.01067 (9)	0.00424 (9)
S1	0.0264 (2)	0.0325 (3)	0.0236 (2)	0.00823 (19)	−0.00028 (17)	−0.00524 (18)
O1	0.0345 (7)	0.0306 (8)	0.0276 (7)	0.0047 (6)	−0.0014 (6)	−0.0037 (6)
O2	0.0379 (7)	0.0294 (8)	0.0352 (8)	0.0090 (6)	−0.0061 (6)	−0.0033 (6)
O3	0.0309 (7)	0.0511 (11)	0.0329 (8)	0.0049 (7)	0.0072 (6)	−0.0131 (7)
C1	0.0293 (9)	0.0319 (11)	0.0229 (9)	0.0079 (8)	−0.0007 (7)	−0.0030 (7)
C2	0.0272 (9)	0.0315 (11)	0.0228 (9)	0.0105 (8)	−0.0028 (7)	−0.0035 (7)
C3	0.0295 (9)	0.0333 (12)	0.0276 (9)	0.0086 (8)	−0.0002 (7)	−0.0019 (8)
C4	0.0314 (9)	0.0382 (12)	0.0255 (9)	0.0132 (8)	0.0027 (7)	0.0025 (8)
C5	0.0359 (10)	0.0439 (13)	0.0230 (9)	0.0166 (9)	−0.0016 (8)	−0.0055 (8)
C6	0.0328 (10)	0.0336 (12)	0.0288 (10)	0.0137 (8)	−0.0049 (8)	−0.0061 (8)
C7	0.0295 (9)	0.0304 (11)	0.0253 (9)	0.0100 (8)	−0.0033 (7)	−0.0012 (8)
C8	0.0317 (9)	0.0304 (11)	0.0271 (9)	0.0100 (8)	−0.0012 (7)	−0.0028 (8)
C9	0.0484 (12)	0.0394 (13)	0.0332 (11)	0.0111 (10)	−0.0072 (9)	−0.0118 (9)
C10	0.0409 (11)	0.0353 (13)	0.0343 (11)	0.0029 (9)	0.0052 (9)	−0.0013 (9)
C11	0.0283 (9)	0.0390 (11)	0.0174 (8)	0.0115 (8)	0.0025 (7)	−0.0011 (7)
C12	0.0287 (9)	0.0419 (13)	0.0239 (9)	0.0067 (8)	0.0044 (7)	−0.0019 (8)
C13	0.0285 (10)	0.0651 (16)	0.0235 (9)	0.0108 (10)	0.0057 (8)	−0.0055 (10)
C14	0.0348 (11)	0.0774 (19)	0.0253 (10)	0.0247 (12)	0.0005 (8)	0.0031 (11)
C15	0.0542 (13)	0.0508 (15)	0.0275 (10)	0.0248 (12)	0.0033 (10)	0.0101 (10)
C16	0.0405 (11)	0.0430 (13)	0.0266 (9)	0.0113 (9)	0.0050 (8)	0.0036 (9)
C17	0.0263 (10)	0.086 (2)	0.0482 (14)	0.0016 (12)	0.0042 (9)	−0.0118 (14)

Geometric parameters (Å, º)

Br1—C4	1.903 (2)	C9—H9A	0.9600
Br1—O2^{i}	3.3350 (16)	C9—H9B	0.9600
S1—O2	1.4341 (17)	C9—H9C	0.9600
S1—O3	1.4400 (15)	C10—H10A	0.9600
S1—C1	1.735 (2)	C10—H10B	0.9600
S1—C11	1.7687 (19)	C10—H10C	0.9600
O1—C8	1.368 (2)	C11—C12	1.381 (3)
O1—C7	1.379 (3)	C11—C16	1.383 (3)
C1—C8	1.358 (3)	C12—C13	1.398 (3)
C1—C2	1.441 (3)	C12—H12	0.9300
C2—C7	1.387 (3)	C13—C14	1.382 (4)
C2—C3	1.391 (3)	C13—C17	1.510 (4)
C3—C4	1.381 (3)	C14—C15	1.379 (4)
C3—H3	0.9300	C14—H14	0.9300
C4—C5	1.394 (3)	C15—C16	1.385 (3)
C5—C6	1.392 (3)	C15—H15	0.9300

C5—H5	0.9300	C16—H16	0.9300
C6—C7	1.385 (3)	C17—H17A	0.9600
C6—C9	1.495 (3)	C17—H17B	0.9600
C8—C10	1.481 (3)	C17—H17C	0.9600
C4—Br1—O2^{i}	168.67 (6)	H9A—C9—H9B	109.5
O2—S1—O3	118.89 (9)	C6—C9—H9C	109.5
O2—S1—C1	108.10 (9)	H9A—C9—H9C	109.5
O3—S1—C1	108.30 (10)	H9B—C9—H9C	109.5
O2—S1—C11	107.76 (10)	C8—C10—H10A	109.5
O3—S1—C11	107.20 (9)	C8—C10—H10B	109.5
C1—S1—C11	105.88 (9)	H10A—C10—H10B	109.5
C8—O1—C7	106.91 (16)	C8—C10—H10C	109.5
C8—C1—C2	107.93 (17)	H10A—C10—H10C	109.5
C8—C1—S1	126.84 (15)	H10B—C10—H10C	109.5
C2—C1—S1	125.22 (16)	C12—C11—C16	122.29 (19)
C7—C2—C3	119.64 (18)	C12—C11—S1	117.95 (16)
C7—C2—C1	104.47 (18)	C16—C11—S1	119.54 (17)
C3—C2—C1	135.88 (19)	C11—C12—C13	119.5 (2)
C4—C3—C2	115.9 (2)	C11—C12—H12	120.2
C4—C3—H3	122.0	C13—C12—H12	120.2
C2—C3—H3	122.0	C14—C13—C12	118.1 (2)
C3—C4—C5	123.7 (2)	C14—C13—C17	122.1 (2)
C3—C4—Br1	118.26 (17)	C12—C13—C17	119.8 (2)
C5—C4—Br1	117.99 (15)	C15—C14—C13	121.8 (2)
C6—C5—C4	120.94 (18)	C15—C14—H14	119.1
C6—C5—H5	119.5	C13—C14—H14	119.1
C4—C5—H5	119.5	C14—C15—C16	120.5 (2)
C7—C6—C5	114.4 (2)	C14—C15—H15	119.8
C7—C6—C9	122.0 (2)	C16—C15—H15	119.8
C5—C6—C9	123.63 (19)	C11—C16—C15	117.8 (2)
O1—C7—C6	124.08 (19)	C11—C16—H16	121.1
O1—C7—C2	110.57 (16)	C15—C16—H16	121.1
C6—C7—C2	125.3 (2)	C13—C17—H17A	109.5
C1—C8—O1	110.12 (17)	C13—C17—H17B	109.5
C1—C8—C10	135.10 (19)	H17A—C17—H17B	109.5
O1—C8—C10	114.78 (19)	C13—C17—H17C	109.5
C6—C9—H9A	109.5	H17A—C17—H17C	109.5
C6—C9—H9B	109.5	H17B—C17—H17C	109.5
O2—S1—C1—C8	−149.32 (17)	C3—C2—C7—O1	179.96 (16)
O3—S1—C1—C8	−19.3 (2)	C1—C2—C7—O1	0.1 (2)
C11—S1—C1—C8	95.42 (18)	C3—C2—C7—C6	2.0 (3)
O2—S1—C1—C2	32.05 (18)	C1—C2—C7—C6	−177.80 (18)
O3—S1—C1—C2	162.10 (16)	C2—C1—C8—O1	0.2 (2)
C11—S1—C1—C2	−83.20 (18)	S1—C1—C8—O1	−178.57 (14)
C8—C1—C2—C7	−0.2 (2)	C2—C1—C8—C10	−179.5 (2)
S1—C1—C2—C7	178.62 (14)	S1—C1—C8—C10	1.7 (3)

C8—C1—C2—C3	180.0 (2)	C7—O1—C8—C1	−0.2 (2)
S1—C1—C2—C3	−1.2 (3)	C7—O1—C8—C10	179.60 (16)
C7—C2—C3—C4	−1.2 (3)	O2—S1—C11—C12	12.15 (17)
C1—C2—C3—C4	178.6 (2)	O3—S1—C11—C12	−116.91 (15)
C2—C3—C4—C5	0.1 (3)	C1—S1—C11—C12	127.64 (16)
C2—C3—C4—Br1	−179.53 (13)	O2—S1—C11—C16	−173.10 (15)
O2^{i}—Br1—C4—C3	58.1 (4)	O3—S1—C11—C16	57.84 (18)
O2^{i}—Br1—C4—C5	−121.5 (3)	C1—S1—C11—C16	−57.62 (18)
C3—C4—C5—C6	0.4 (3)	C16—C11—C12—C13	−1.1 (3)
Br1—C4—C5—C6	−179.97 (15)	S1—C11—C12—C13	173.54 (15)
C4—C5—C6—C7	0.2 (3)	C11—C12—C13—C14	1.2 (3)
C4—C5—C6—C9	−178.61 (19)	C11—C12—C13—C17	−178.06 (19)
C8—O1—C7—C6	177.97 (18)	C12—C13—C14—C15	−0.6 (3)
C8—O1—C7—C2	0.0 (2)	C17—C13—C14—C15	178.7 (2)
C5—C6—C7—O1	−179.12 (17)	C13—C14—C15—C16	−0.3 (3)
C9—C6—C7—O1	−0.3 (3)	C12—C11—C16—C15	0.2 (3)
C5—C6—C7—C2	−1.5 (3)	S1—C11—C16—C15	−174.34 (15)
C9—C6—C7—C2	177.39 (19)	C14—C15—C16—C11	0.5 (3)

Symmetry code: (i) −*x*+1, −*y*+2, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10*B*···O2^{ii}	0.96	2.54	3.338 (3)	141
C17—H17*C*···O3^{iii}	0.96	2.41	3.357 (3)	170

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