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3-(4-Bromophenylsulfinyl)-2,5,7-trimethyl-1-benzofuran

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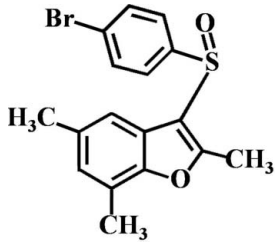
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.086; data-to-parameter ratio = 19.8.

In the title compound, $C_{17}H_{15}BrO_2S$, the 4-bromophenyl ring makes a dihedral angle of 87.78 (5)° with the mean plane of the benzofuran fragment. In the crystal, molecules are linked by weak C—H···O hydrogen bonds, and by weak intermolecular C—S···π [3.399 (2) Å] and C—Br···π [3.797 (2) and 3.757 (2) Å] interactions.

Related literature

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



Experimental

Crystal data

$C_{17}H_{15}BrO_2S$
M_r = 363.26
Triclinic, $P\overline{1}$
a = 6.1034 (1) Å
b = 10.2278 (2) Å
c = 12.6731 (2) Å
α = 84.586 (1)°
β = 79.419 (1)°
γ = 85.730 (1)°
V = 772.87 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.80 mm^{-1}
T = 173 K
0.37 × 0.34 × 0.25 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.474, T_{max} = 0.746
14146 measured reflections
3826 independent reflections
3319 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.086
S = 1.06
3826 reflections
193 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.83 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13···O2^{i}	0.95	2.50	3.233 (2)	134

Symmetry code: (i) $-x+2, -y+1, -z+1$.

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* (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: PV2507).

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Choi, H. D., Seo, P. J., Son, B. W. & Lee, U. (2010*b*). *Acta Cryst.* E**66**, o2325.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
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supporting information

Acta Cryst. (2012). E**68**, o482 [doi:10.1107/S1600536812001523]

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S1. Comment

As a part of our continuing study of 2,5,7-trimethyl-1-benzofuran derivatives containing 3-(4-fluorophenylsulfinyl) (Choi *et al.*, 2010*a*) and 3-(4-chlorophenylsufinyl) (Choi *et al.*, 2010*b*) substituents, 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 0.007 (1) Å from the least-squares plane defined by the nine constituent atoms. The dihedral angle between the 4-bromophenyl ring and the mean plane of the benzofurn fragment is 87.78 (5)°. The crystal packing (Fig. 2) is stabilized by weak intermolecular C13—H13···O2 hydrogen bonds (Table 1). The crystal packing is further stabilized by intermolecular C15—Br1···π interactions between the bromine atom and the benzene rings of a neighbouring molecule with Br1···Cg1^{iii} and Br1···Cg2^{iii} being 3.757 (2) and 3.797 (2) Å, respectively. (Cg1 and Cg2 are the centroid of the C12–C17 and C2–C7 benzene rings, respectively.) In addition, there is a weak intermolecular S···π interaction between the sulfur and the centroid of the benzene ring (C12–C17) of an adjacent molecule, with S1···Cg1^{ii} 3.399 (2) Å.

S2. Experimental

77% 3-Chloroperoxybenzoic acid (224 mg, 0.9 mmol) was added in small portions to a stirred solution of 3-(4-bromophenylsulfanyl)-2,5,7-trimethyl-1-benzofuran (312 mg, 0.9 mmol) in dichloromethane (40 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, 2:1 v/v) to afford the title compound as a colorless solid [yield 71%, m.p. 431–432 K; R_f = 0.53 (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 ethyl acetate at room temperature.

S3. Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95 and 0.98 Å for aryl and methyl H atoms, respectively, and U_{iso}(H) = 1.2U_{eq}(C) for aryl and 1.5U_{eq}(C) for methyl H atoms.

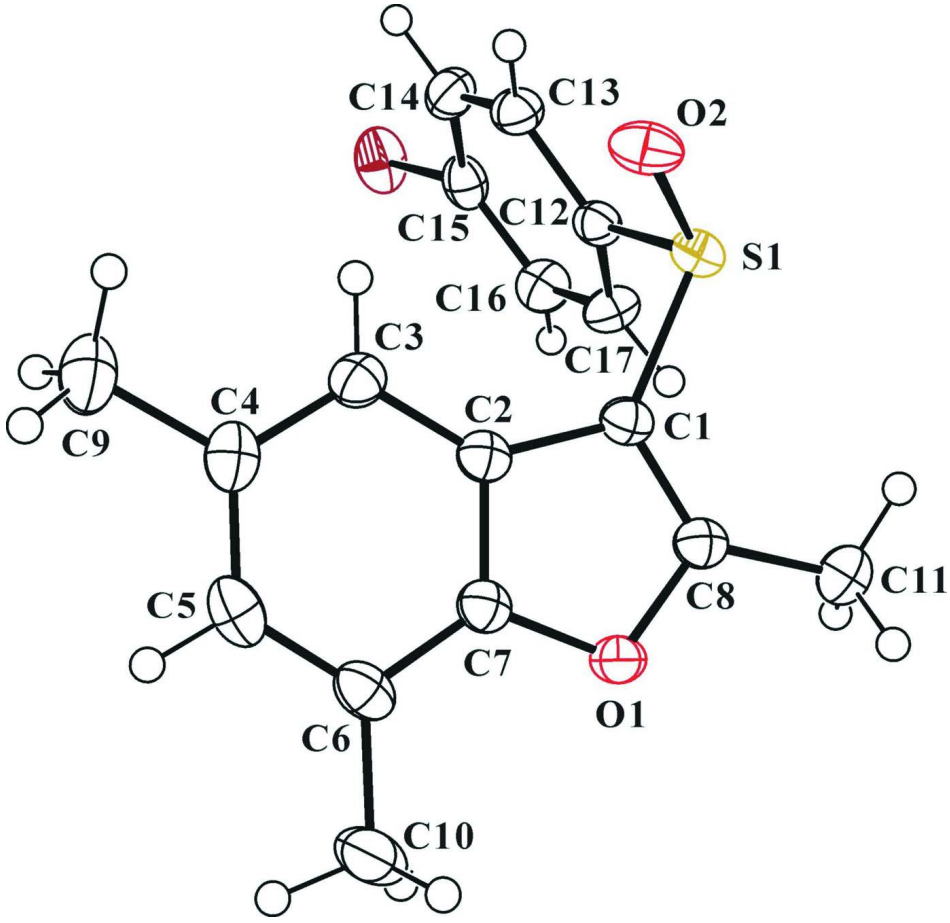

Figure 1

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 radii.

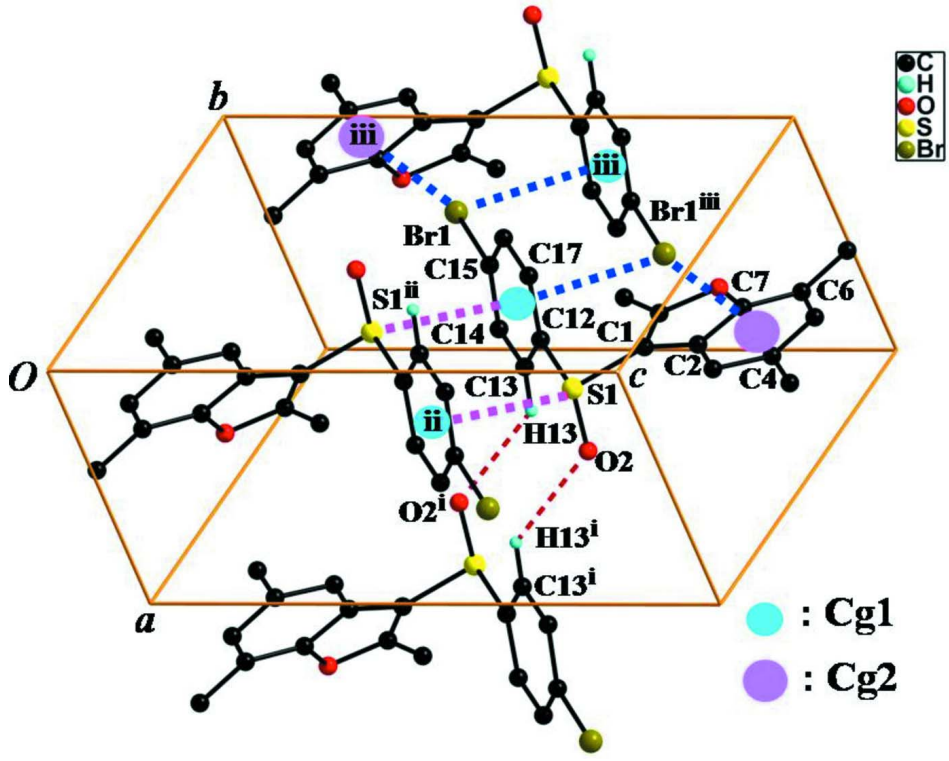

Figure 2

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

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γ = 85.730 (1)°
V = 772.87 (2) Å^3
Z = 2
F(000) = 368
D_x = 1.561 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8427 reflections
θ = 2.5–28.2°
μ = 2.80 mm^{-1}
T = 173 K
Block, colourless
0.37 × 0.34 × 0.25 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} = 0.474, T_{max} = 0.746
14146 measured reflections
3826 independent reflections
3319 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

θ_{max} = 28.3°, θ_{min} = 1.6°
h = −8→8
k = −13→13
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.086
S = 1.06
3826 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
$w = 1/[\sigma^2(F_o^2) + (0.0446P)^2 + 0.243P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.46 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 $F^2 > 2\text{sigma}(F^2)$ 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	0.40055 (4)	0.990460 (19)	0.338480 (16)	0.04068 (9)
S1	0.60888 (8)	0.48980 (4)	0.65970 (3)	0.02498 (11)
O1	0.2929 (2)	0.59852 (12)	0.94015 (10)	0.0265 (3)
O2	0.8568 (2)	0.47089 (14)	0.64796 (11)	0.0343 (3)
C1	0.5094 (3)	0.56250 (17)	0.78141 (14)	0.0237 (4)
C2	0.6083 (3)	0.66325 (16)	0.82636 (14)	0.0237 (4)
C3	0.7953 (3)	0.73737 (17)	0.79577 (15)	0.0272 (4)
H3	0.8927	0.7269	0.7291	0.033*
C4	0.8367 (3)	0.82671 (18)	0.86439 (16)	0.0306 (4)
C5	0.6874 (4)	0.84262 (18)	0.96147 (16)	0.0329 (4)
H5	0.7167	0.9058	1.0067	0.039*
C6	0.4990 (3)	0.77049 (18)	0.99471 (14)	0.0297 (4)
C7	0.4683 (3)	0.68124 (17)	0.92437 (14)	0.0250 (4)
C8	0.3247 (3)	0.52683 (17)	0.85186 (14)	0.0249 (4)
C9	1.0408 (4)	0.9067 (2)	0.8340 (2)	0.0417 (5)
H9A	1.1561	0.8592	0.7851	0.063*
H9B	1.0982	0.9209	0.8990	0.063*
H9C	1.0006	0.9918	0.7980	0.063*
C10	0.3388 (4)	0.7868 (2)	1.09857 (16)	0.0422 (6)
H10A	0.1892	0.8123	1.0831	0.063*
H10B	0.3879	0.8553	1.1366	0.063*
H10C	0.3348	0.7035	1.1437	0.063*
C11	0.1536 (3)	0.43163 (19)	0.85082 (16)	0.0318 (4)

H11A	0.0141	0.4795	0.8386	0.048*
H11B	0.1275	0.3789	0.9202	0.048*
H11C	0.2061	0.3736	0.7930	0.048*
C13	0.7261 (3)	0.68142 (19)	0.49533 (15)	0.0277 (4)
H13	0.8740	0.6436	0.4918	0.033*
C12	0.5531 (3)	0.63048 (17)	0.57012 (14)	0.0232 (4)
C14	0.6811 (4)	0.78872 (19)	0.42530 (15)	0.0311 (4)
H14	0.7981	0.8255	0.3736	0.037*
C15	0.4645 (4)	0.84151 (18)	0.43155 (15)	0.0283 (4)
C16	0.2898 (3)	0.78812 (19)	0.50419 (16)	0.0309 (4)
H16	0.1415	0.8247	0.5064	0.037*
C17	0.3341 (3)	0.68074 (19)	0.57347 (16)	0.0303 (4)
H17	0.2160	0.6416	0.6229	0.036*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.05809 (17)	0.03134 (12)	0.03613 (13)	−0.00576 (10)	−0.01923 (11)	0.00288 (8)
S1	0.0259 (2)	0.0250 (2)	0.0236 (2)	0.00246 (17)	−0.00289 (18)	−0.00540 (16)
O1	0.0265 (7)	0.0282 (6)	0.0229 (6)	−0.0006 (5)	0.0002 (5)	−0.0031 (5)
O2	0.0269 (7)	0.0430 (8)	0.0315 (7)	0.0107 (6)	−0.0044 (6)	−0.0077 (6)
C1	0.0244 (9)	0.0237 (8)	0.0222 (8)	0.0019 (7)	−0.0029 (7)	−0.0025 (6)
C2	0.0236 (9)	0.0224 (8)	0.0247 (8)	0.0032 (7)	−0.0050 (7)	−0.0012 (6)
C3	0.0227 (9)	0.0264 (8)	0.0314 (9)	0.0018 (7)	−0.0033 (7)	−0.0015 (7)
C4	0.0292 (10)	0.0237 (8)	0.0410 (11)	−0.0006 (7)	−0.0136 (8)	0.0012 (7)
C5	0.0439 (12)	0.0239 (8)	0.0347 (10)	0.0021 (8)	−0.0171 (9)	−0.0054 (7)
C6	0.0394 (12)	0.0252 (8)	0.0252 (9)	0.0047 (8)	−0.0093 (8)	−0.0037 (7)
C7	0.0273 (10)	0.0225 (8)	0.0245 (8)	0.0015 (7)	−0.0046 (7)	−0.0012 (6)
C8	0.0253 (9)	0.0248 (8)	0.0237 (8)	0.0016 (7)	−0.0037 (7)	−0.0010 (6)
C9	0.0341 (12)	0.0329 (10)	0.0608 (14)	−0.0058 (9)	−0.0138 (10)	−0.0044 (9)
C10	0.0604 (16)	0.0365 (11)	0.0277 (10)	0.0027 (10)	−0.0013 (10)	−0.0096 (8)
C11	0.0286 (10)	0.0324 (9)	0.0344 (10)	−0.0059 (8)	−0.0049 (8)	−0.0003 (8)
C13	0.0216 (9)	0.0333 (9)	0.0281 (9)	−0.0024 (7)	−0.0022 (7)	−0.0067 (7)
C12	0.0232 (9)	0.0261 (8)	0.0211 (8)	−0.0014 (7)	−0.0039 (7)	−0.0057 (6)
C14	0.0318 (11)	0.0343 (10)	0.0271 (9)	−0.0096 (8)	−0.0021 (8)	−0.0014 (7)
C15	0.0367 (11)	0.0255 (8)	0.0257 (9)	−0.0038 (8)	−0.0118 (8)	−0.0033 (7)
C16	0.0251 (10)	0.0344 (10)	0.0342 (10)	0.0017 (8)	−0.0091 (8)	−0.0028 (8)
C17	0.0229 (10)	0.0354 (10)	0.0300 (9)	−0.0012 (8)	0.0005 (8)	−0.0006 (8)

Geometric parameters (Å, º)

Br1—C15	1.8988 (19)	C9—H9A	0.9800
S1—O2	1.4929 (15)	C9—H9B	0.9800
S1—C1	1.7623 (17)	C9—H9C	0.9800
S1—C12	1.7989 (18)	C10—H10A	0.9800
O1—C8	1.372 (2)	C10—H10B	0.9800
O1—C7	1.388 (2)	C10—H10C	0.9800
C1—C8	1.353 (3)	C11—H11A	0.9800

C1—C2	1.442 (3)	C11—H11B	0.9800
C2—C7	1.392 (2)	C11—H11C	0.9800
C2—C3	1.392 (3)	C13—C12	1.381 (3)
C3—C4	1.386 (3)	C13—C14	1.389 (3)
C3—H3	0.9500	C13—H13	0.9500
C4—C5	1.405 (3)	C12—C17	1.390 (3)
C4—C9	1.512 (3)	C14—C15	1.382 (3)
C5—C6	1.391 (3)	C14—H14	0.9500
C5—H5	0.9500	C15—C16	1.383 (3)
C6—C7	1.380 (3)	C16—C17	1.382 (3)
C6—C10	1.504 (3)	C16—H16	0.9500
C8—C11	1.482 (3)	C17—H17	0.9500

O2—S1—C1	107.54 (9)	H9A—C9—H9C	109.5
O2—S1—C12	106.52 (9)	H9B—C9—H9C	109.5
C1—S1—C12	97.22 (8)	C6—C10—H10A	109.5
C8—O1—C7	106.40 (13)	C6—C10—H10B	109.5
C8—C1—C2	107.98 (15)	H10A—C10—H10B	109.5
C8—C1—S1	124.04 (14)	C6—C10—H10C	109.5
C2—C1—S1	127.94 (14)	H10A—C10—H10C	109.5
C7—C2—C3	119.24 (17)	H10B—C10—H10C	109.5
C7—C2—C1	104.52 (16)	C8—C11—H11A	109.5
C3—C2—C1	136.24 (16)	C8—C11—H11B	109.5
C4—C3—C2	118.80 (17)	H11A—C11—H11B	109.5
C4—C3—H3	120.6	C8—C11—H11C	109.5
C2—C3—H3	120.6	H11A—C11—H11C	109.5
C3—C4—C5	119.54 (18)	H11B—C11—H11C	109.5
C3—C4—C9	119.84 (19)	C12—C13—C14	119.09 (18)
C5—C4—C9	120.62 (19)	C12—C13—H13	120.5
C6—C5—C4	123.30 (18)	C14—C13—H13	120.5
C6—C5—H5	118.3	C13—C12—C17	121.34 (17)
C4—C5—H5	118.3	C13—C12—S1	119.43 (14)
C7—C6—C5	114.68 (17)	C17—C12—S1	119.12 (14)
C7—C6—C10	121.73 (19)	C15—C14—C13	119.36 (18)
C5—C6—C10	123.59 (18)	C15—C14—H14	120.3
C6—C7—O1	125.11 (16)	C13—C14—H14	120.3
C6—C7—C2	124.42 (18)	C14—C15—C16	121.62 (18)
O1—C7—C2	110.46 (16)	C14—C15—Br1	119.99 (15)
C1—C8—O1	110.62 (16)	C16—C15—Br1	118.39 (15)
C1—C8—C11	133.50 (17)	C17—C16—C15	119.08 (18)
O1—C8—C11	115.86 (15)	C17—C16—H16	120.5
C4—C9—H9A	109.5	C15—C16—H16	120.5
C4—C9—H9B	109.5	C16—C17—C12	119.41 (18)
H9A—C9—H9B	109.5	C16—C17—H17	120.3
C4—C9—H9C	109.5	C12—C17—H17	120.3

O2—S1—C1—C8	−137.92 (16)	C1—C2—C7—C6	−179.37 (17)
C12—S1—C1—C8	112.17 (17)	C3—C2—C7—O1	−179.85 (15)

O2—S1—C1—C2	39.33 (18)	C1—C2—C7—O1	−0.31 (19)
C12—S1—C1—C2	−70.58 (17)	C2—C1—C8—O1	0.7 (2)
C8—C1—C2—C7	−0.3 (2)	S1—C1—C8—O1	178.45 (12)
S1—C1—C2—C7	−177.85 (14)	C2—C1—C8—C11	178.9 (2)
C8—C1—C2—C3	179.2 (2)	S1—C1—C8—C11	−3.4 (3)
S1—C1—C2—C3	1.6 (3)	C7—O1—C8—C1	−0.92 (19)
C7—C2—C3—C4	0.3 (3)	C7—O1—C8—C11	−179.41 (15)
C1—C2—C3—C4	−179.09 (19)	C14—C13—C12—C17	2.9 (3)
C2—C3—C4—C5	−1.5 (3)	C14—C13—C12—S1	178.98 (14)
C2—C3—C4—C9	178.93 (18)	O2—S1—C12—C13	11.31 (17)
C3—C4—C5—C6	1.5 (3)	C1—S1—C12—C13	122.07 (15)
C9—C4—C5—C6	−178.92 (19)	O2—S1—C12—C17	−172.47 (15)
C4—C5—C6—C7	−0.2 (3)	C1—S1—C12—C17	−61.72 (17)
C4—C5—C6—C10	179.87 (19)	C12—C13—C14—C15	−0.4 (3)
C5—C6—C7—O1	179.99 (16)	C13—C14—C15—C16	−1.6 (3)
C10—C6—C7—O1	−0.1 (3)	C13—C14—C15—Br1	178.71 (14)
C5—C6—C7—C2	−1.1 (3)	C14—C15—C16—C17	1.2 (3)
C10—C6—C7—C2	178.82 (18)	Br1—C15—C16—C17	−179.15 (15)
C8—O1—C7—C6	179.80 (17)	C15—C16—C17—C12	1.3 (3)
C8—O1—C7—C2	0.75 (19)	C13—C12—C17—C16	−3.3 (3)
C3—C2—C7—C6	1.1 (3)	S1—C12—C17—C16	−179.44 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13···O2^{i}	0.95	2.50	3.233 (2)	134

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