Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-(4-Bromophenylsulfinyl)-5-chloro-2,7-dimethyl-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

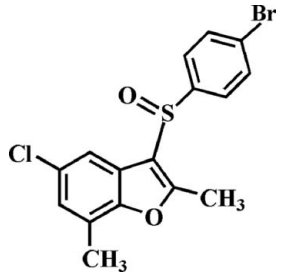
Received 3 April 2013; accepted 11 April 2013

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.031; wR factor = 0.082; data-to-parameter ratio = 19.8.

In the title compound, $C_{16}H_{12}BrClO_2S$, the 4-bromophenyl ring makes a dihedral angle of 88.84 (5)° with the mean plane [r.m.s. deviation = 0.009 (1) Å] of the benzofuran fragment. In the crystal, molecules are linked by weak C—H···O and C—S···π [3.386 (2) Å] interactions, forming a chain perpendicular to the *bc* plane.

Related literature

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



Experimental

Crystal data

$C_{16}H_{12}BrClO_2S$
M_r = 383.68
Triclinic, $P\overline{1}$
a = 6.1266 (3) Å
b = 10.0247 (5) Å
c = 12.6630 (7) Å
α = 84.749 (3)°
β = 79.235 (2)°
γ = 86.443 (3)°
V = 760.03 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 3.02 mm^{-1}
T = 173 K
0.33 × 0.23 × 0.16 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.506, T_{max} = 0.746
13853 measured reflections
3794 independent reflections
3209 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.082
S = 1.05
3794 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.72 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···O2^{i}	0.95	2.50	3.249 (2)	136

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

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* for Windows (Farrugia, 2012) and *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *SHELXL97*.

This work was supported by the Blue-Bio Industry Regional Innovation Center (RIC08-06-07) at Dongeui University as an RIC program under the Ministry of Knowledge Economy and Busan city.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AA2089).

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*a*). *Acta Cryst.* E**68**, o2027.
Choi, H. D., Seo, P. J. & Lee, U. (2012*b*). *Acta Cryst.* E**68**, o2080.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2013). E**69**, o745 [https://doi.org/10.1107/S160053681300994X]

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

Hong Dae Choi, Pil Ja Seo and Uk Lee

S1. Comment

As a part of our continuing study of 5-chloro-2-methyl-1-benzofuran derivatives containing 4-bromophenylsulfonyl (Choi *et al.*, 2012*a*) and 4-bromophenylsulfinyl (Choi *et al.*, 2012*b*) 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 0.009 (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 benzofuran ring is 88.84 (5)°. In the crystal structure (Fig. 2), molecules are connected by weak C—H···O hydrogen bonds (Table 1), and by intermolecular C—S···π interactions between the sulfur atom and the 4-bromophenyl ring of an adjacent molecule, with a S1···Cg^{ii} being 3.386 (2) Å (Cg is the centroid of the C11/C16 ring).

S2. Experimental

3-Chloroperoxybenzoic acid (77%, 224 mg, 1.0 mmol) was added in small portions to a stirred solution of 3-(4-bromophenylsulfanyl)-5-chloro-2,7-dimethyl-1-benzofuran (331 mg, 0.9 mmol) in dichloromethane (30 mL) at 273 K. After being stirred at room temperature for 5 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 79%, m.p. 442–443 K; R_f = 0.78 (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 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.

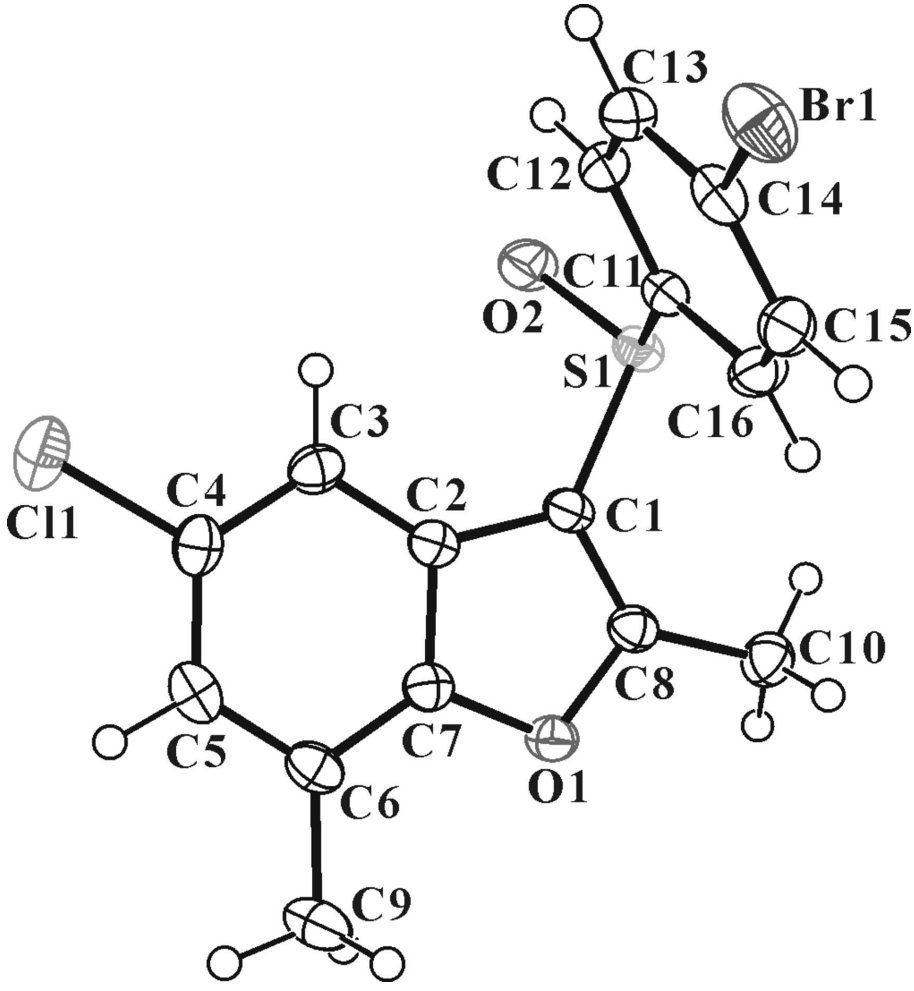


Figure 1

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.

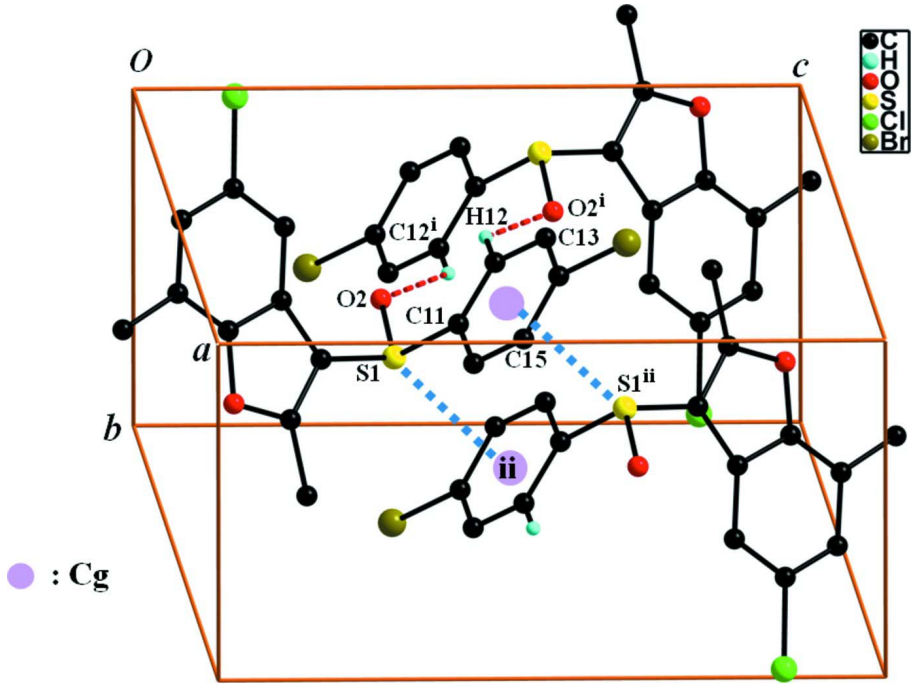


Figure 2

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

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

Crystal data

$C_{16}H_{12}BrClO_2S$
M_r = 383.68
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 6.1266 (3) Å
b = 10.0247 (5) Å
c = 12.6630 (7) Å
α = 84.749 (3)°
β = 79.235 (2)°
γ = 86.443 (3)°
V = 760.03 (7) Å^3
Z = 2
$F(000)$ = 384
D_x = 1.677 Mg m^{-3}
Melting point = 442–443 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6958 reflections
θ = 2.5–28.5°
μ = 3.02 mm^{-1}
T = 173 K
Block, colourless
0.33 × 0.23 × 0.16 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} = 0.506, T_{max} = 0.746
13853 measured reflections
3794 independent reflections
3209 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 28.5°, θ_{min} = 1.6°
h = −8→8
k = −13→13
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.082$
$S = 1.05$
3794 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
$w = 1/[\sigma^2(F_o^2) + (0.0396P)^2 + 0.2147P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.72$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.60695 (4)	0.00462 (2)	0.662469 (16)	0.03820 (9)
Cl1	−0.07539 (9)	0.08106 (6)	0.16204 (5)	0.03950 (14)
S1	0.38584 (8)	0.50904 (4)	0.34160 (3)	0.02363 (11)
O1	0.7066 (2)	0.40065 (13)	0.06063 (10)	0.0251 (3)
O2	0.1390 (2)	0.52341 (15)	0.35333 (11)	0.0326 (3)
C1	0.4874 (3)	0.43659 (18)	0.21924 (14)	0.0226 (4)
C2	0.3886 (3)	0.33547 (18)	0.17248 (14)	0.0218 (4)
C3	0.1985 (3)	0.26119 (19)	0.20141 (15)	0.0253 (4)
H3	0.0981	0.2709	0.2674	0.030*
C4	0.1635 (3)	0.17304 (19)	0.12968 (16)	0.0266 (4)
C5	0.3103 (3)	0.15431 (19)	0.03288 (15)	0.0288 (4)
H5	0.2794	0.0908	−0.0130	0.035*
C6	0.5011 (3)	0.22733 (19)	0.00283 (14)	0.0266 (4)
C7	0.5301 (3)	0.31709 (18)	0.07481 (14)	0.0228 (4)
C8	0.6746 (3)	0.47297 (19)	0.14948 (14)	0.0235 (4)
C9	0.6643 (4)	0.2110 (2)	−0.10013 (16)	0.0366 (5)
H9A	0.6792	0.2976	−0.1429	0.055*
H9B	0.6108	0.1458	−0.1414	0.055*
H9C	0.8092	0.1791	−0.0832	0.055*
C10	0.8461 (3)	0.5696 (2)	0.15219 (16)	0.0301 (4)
H10A	0.7906	0.6308	0.2083	0.045*
H10B	0.8800	0.6211	0.0820	0.045*
H10C	0.9813	0.5208	0.1681	0.045*
C11	0.4447 (3)	0.36641 (18)	0.43057 (14)	0.0219 (4)
C12	0.2744 (3)	0.3133 (2)	0.50722 (15)	0.0258 (4)

H12	0.1267	0.3504	0.5121	0.031*
C13	0.3225 (3)	0.2049 (2)	0.57709 (15)	0.0290 (4)
H13	0.2075	0.1662	0.6297	0.035*
C14	0.5385 (4)	0.15413 (19)	0.56925 (15)	0.0267 (4)
C15	0.7110 (3)	0.2096 (2)	0.49468 (16)	0.0292 (4)
H15	0.8592	0.1738	0.4912	0.035*
C16	0.6637 (3)	0.3177 (2)	0.42569 (16)	0.0285 (4)
H16	0.7799	0.3585	0.3752	0.034*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.05595 (17)	0.03092 (13)	0.03054 (13)	−0.00131 (10)	−0.01697 (10)	0.00087 (8)
Cl1	0.0293 (3)	0.0376 (3)	0.0541 (3)	−0.0073 (2)	−0.0115 (2)	−0.0046 (2)
S1	0.0255 (2)	0.0252 (2)	0.0202 (2)	0.00279 (19)	−0.00371 (18)	−0.00560 (17)
O1	0.0249 (7)	0.0283 (7)	0.0209 (6)	−0.0009 (6)	−0.0012 (5)	−0.0026 (5)
O2	0.0245 (7)	0.0433 (8)	0.0296 (7)	0.0106 (6)	−0.0056 (6)	−0.0079 (6)
C1	0.0237 (9)	0.0257 (9)	0.0181 (8)	0.0025 (8)	−0.0034 (7)	−0.0038 (7)
C2	0.0227 (9)	0.0225 (8)	0.0201 (8)	0.0033 (7)	−0.0050 (7)	−0.0013 (7)
C3	0.0214 (9)	0.0266 (9)	0.0266 (9)	0.0029 (8)	−0.0026 (7)	−0.0012 (7)
C4	0.0249 (10)	0.0228 (9)	0.0336 (10)	−0.0008 (8)	−0.0105 (8)	−0.0002 (7)
C5	0.0364 (11)	0.0251 (9)	0.0278 (9)	0.0004 (8)	−0.0127 (8)	−0.0051 (7)
C6	0.0330 (11)	0.0259 (9)	0.0212 (9)	0.0044 (8)	−0.0069 (8)	−0.0029 (7)
C7	0.0229 (9)	0.0240 (9)	0.0211 (8)	0.0010 (7)	−0.0047 (7)	−0.0002 (7)
C8	0.0247 (9)	0.0253 (9)	0.0202 (8)	0.0013 (8)	−0.0045 (7)	−0.0014 (7)
C9	0.0482 (14)	0.0354 (11)	0.0245 (10)	−0.0002 (10)	0.0000 (9)	−0.0088 (8)
C10	0.0284 (10)	0.0311 (10)	0.0305 (10)	−0.0049 (9)	−0.0046 (8)	−0.0003 (8)
C11	0.0229 (9)	0.0257 (9)	0.0178 (8)	−0.0010 (7)	−0.0040 (7)	−0.0051 (7)
C12	0.0193 (9)	0.0328 (10)	0.0255 (9)	−0.0011 (8)	−0.0021 (7)	−0.0075 (8)
C13	0.0301 (11)	0.0334 (10)	0.0232 (9)	−0.0079 (9)	−0.0012 (8)	−0.0032 (8)
C14	0.0357 (11)	0.0247 (9)	0.0220 (9)	−0.0024 (8)	−0.0097 (8)	−0.0036 (7)
C15	0.0234 (10)	0.0351 (10)	0.0296 (10)	0.0038 (8)	−0.0073 (8)	−0.0044 (8)
C16	0.0218 (9)	0.0355 (11)	0.0263 (9)	0.0015 (8)	−0.0010 (8)	−0.0014 (8)

Geometric parameters (Å, º)

Br1—C14	1.8972 (19)	C8—C10	1.480 (3)
Cl1—C4	1.742 (2)	C9—H9A	0.9800
S1—O2	1.4907 (15)	C9—H9B	0.9800
S1—C1	1.7622 (17)	C9—H9C	0.9800
S1—C11	1.7990 (19)	C10—H10A	0.9800
O1—C8	1.371 (2)	C10—H10B	0.9800
O1—C7	1.384 (2)	C10—H10C	0.9800
C1—C8	1.358 (3)	C11—C12	1.382 (3)
C1—C2	1.437 (3)	C11—C16	1.392 (3)
C2—C7	1.392 (2)	C12—C13	1.390 (3)
C2—C3	1.394 (3)	C12—H12	0.9500
C3—C4	1.377 (3)	C13—C14	1.377 (3)

C3—H3	0.9500	C13—H13	0.9500
C4—C5	1.399 (3)	C14—C15	1.386 (3)
C5—C6	1.391 (3)	C15—C16	1.380 (3)
C5—H5	0.9500	C15—H15	0.9500
C6—C7	1.379 (3)	C16—H16	0.9500
C6—C9	1.503 (3)		

O2—S1—C1	107.21 (9)	C6—C9—H9B	109.5
O2—S1—C11	106.44 (9)	H9A—C9—H9B	109.5
C1—S1—C11	97.25 (8)	C6—C9—H9C	109.5
C8—O1—C7	106.52 (13)	H9A—C9—H9C	109.5
C8—C1—C2	107.65 (16)	H9B—C9—H9C	109.5
C8—C1—S1	124.42 (15)	C8—C10—H10A	109.5
C2—C1—S1	127.88 (14)	C8—C10—H10B	109.5
C7—C2—C3	119.18 (18)	H10A—C10—H10B	109.5
C7—C2—C1	104.93 (17)	C8—C10—H10C	109.5
C3—C2—C1	135.88 (17)	H10A—C10—H10C	109.5
C4—C3—C2	116.76 (17)	H10B—C10—H10C	109.5
C4—C3—H3	121.6	C12—C11—C16	121.24 (18)
C2—C3—H3	121.6	C12—C11—S1	119.47 (14)
C3—C4—C5	123.07 (18)	C16—C11—S1	119.13 (14)
C3—C4—Cl1	117.96 (15)	C11—C12—C13	119.01 (18)
C5—C4—Cl1	118.97 (16)	C11—C12—H12	120.5
C6—C5—C4	120.94 (18)	C13—C12—H12	120.5
C6—C5—H5	119.5	C14—C13—C12	119.35 (18)
C4—C5—H5	119.5	C14—C13—H13	120.3
C7—C6—C5	114.94 (16)	C12—C13—H13	120.3
C7—C6—C9	121.98 (19)	C13—C14—C15	121.87 (18)
C5—C6—C9	123.08 (18)	C13—C14—Br1	120.01 (15)
C6—C7—O1	124.64 (16)	C15—C14—Br1	118.11 (15)
C6—C7—C2	125.08 (19)	C16—C15—C14	118.83 (18)
O1—C7—C2	110.28 (16)	C16—C15—H15	120.6
C1—C8—O1	110.62 (17)	C14—C15—H15	120.6
C1—C8—C10	133.30 (17)	C15—C16—C11	119.59 (18)
O1—C8—C10	116.07 (16)	C15—C16—H16	120.2
C6—C9—H9A	109.5	C11—C16—H16	120.2

O2—S1—C1—C8	140.11 (16)	C1—C2—C7—C6	179.24 (18)
C11—S1—C1—C8	−110.13 (17)	C3—C2—C7—O1	179.36 (15)
O2—S1—C1—C2	−37.03 (19)	C1—C2—C7—O1	0.1 (2)
C11—S1—C1—C2	72.73 (18)	C2—C1—C8—O1	−0.9 (2)
C8—C1—C2—C7	0.5 (2)	S1—C1—C8—O1	−178.55 (13)
S1—C1—C2—C7	178.04 (14)	C2—C1—C8—C10	−179.3 (2)
C8—C1—C2—C3	−178.6 (2)	S1—C1—C8—C10	3.1 (3)
S1—C1—C2—C3	−1.1 (3)	C7—O1—C8—C1	0.9 (2)
C7—C2—C3—C4	−0.1 (3)	C7—O1—C8—C10	179.63 (16)
C1—C2—C3—C4	179.0 (2)	O2—S1—C11—C12	−13.18 (18)
C2—C3—C4—C5	1.3 (3)	C1—S1—C11—C12	−123.58 (16)

C2—C3—C4—Cl1	−178.72 (14)	O2—S1—C11—C16	171.25 (15)
C3—C4—C5—C6	−1.2 (3)	C1—S1—C11—C16	60.86 (17)
Cl1—C4—C5—C6	178.83 (14)	C16—C11—C12—C13	−3.2 (3)
C4—C5—C6—C7	−0.2 (3)	S1—C11—C12—C13	−178.65 (15)
C4—C5—C6—C9	179.95 (19)	C11—C12—C13—C14	0.8 (3)
C5—C6—C7—O1	−179.37 (16)	C12—C13—C14—C15	1.2 (3)
C9—C6—C7—O1	0.5 (3)	C12—C13—C14—Br1	−179.12 (15)
C5—C6—C7—C2	1.6 (3)	C13—C14—C15—C16	−0.8 (3)
C9—C6—C7—C2	−178.61 (18)	Br1—C14—C15—C16	179.46 (15)
C8—O1—C7—C6	−179.79 (18)	C14—C15—C16—C11	−1.5 (3)
C8—O1—C7—C2	−0.60 (19)	C12—C11—C16—C15	3.5 (3)
C3—C2—C7—C6	−1.5 (3)	S1—C11—C16—C15	179.03 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···O2^{i}	0.95	2.50	3.249 (2)	136

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