Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-Bromo-3-cyclohexylsulfinyl-2,7-dimethyl-1-benzofuran

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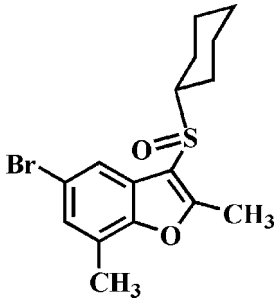
Received 21 March 2011; accepted 27 March 2011

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.030; wR factor = 0.077; data-to-parameter ratio = 21.2.

In the title compound, $C_{16}H_{19}BrO_2S$, the cyclohexyl ring adopts a chair conformation. In the crystal, molecules are linked by a Br···Br [3.5994 (5) Å] contact and a C—H···π interaction involving the phenyl ring of the benzofuran. The crystal structure also exhibits a slipped π–π interaction between the furan rings of neighbouring molecules [centroid–centroid distance = 3.767 (1) Å and interplanar distance of 3.452 (1) Å with a slippage of 1.508 Å].

Related literature

For the pharmacological activity of benzofuran compounds, see: Aslam *et al.* (2006); Galal *et al.* (2009); Khan *et al.* (2005). For natural products with benzofuran rings, see: Akgul & Anil (2003); Soekamto *et al.* (2003). For structural studies of the related 5-bromo-3-cyclohexylsulfinyl-2-methyl-1-benzofuran, see: Choi *et al.* (2011).



Experimental

Crystal data

$C_{16}H_{19}BrO_2S$
M_r = 355.28
Triclinic, $P\overline{1}$
a = 6.3957 (2) Å
b = 11.2576 (3) Å
c = 12.0384 (3) Å
α = 104.726 (2)°
β = 101.426 (1)°
γ = 106.380 (1)°
V = 769.53 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.81 mm^{-1}
T = 173 K
0.19 × 0.17 × 0.09 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.660, T_{max} = 0.746
14554 measured reflections
3887 independent reflections
3165 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.077
S = 1.03
3887 reflections
183 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

*Cg*1 is the centroid of the phenyl ring of the benzofuran.

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10*B*···*Cg*1^{i}	0.98	2.97	3.717 (2)	134

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

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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2669).

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supporting information

Acta Cryst. (2011). E**67**, o1039 [doi:10.1107/S1600536811011354]

5-Bromo-3-cyclohexylsulfinyl-2,7-dimethyl-1-benzofuran

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S1. Comment

Many compounds having a benzofuran skeleton have attracted interesting pharmacological properties such as antifungal, antitumor and antiviral, and antimicrobial activities (Aslam *et al.*, 2006, Galal *et al.*, 2009, Khan *et al.*, 2005). These compounds occur in a wide range of natural products (Akgul & Anil, 2003; Soekamto *et al.*, 2003). As a part of our ongoing study of the substituent effect on the solid state structures of 3-cyclohexylsulfinyl-5-halo-2-methyl-1-benzofuran analogues (Choi *et al.*, 2011), 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 0.010 (1) Å from the least-squares plane defined by the nine constituent atoms. The crystal packing (Fig. 2) is stabilized by an intermolecular Br1···Br1^{i} contact at 3.5994 (5) Å, and by a weak intermolecular C—H···π interaction (Table 1, Cg1 is the centroid of the C2–C7 phenyl ring of the benzofuran). The crystal packing (Fig. 2) is further stabilized by a weak slippest π···π interaction between the furan rings of neighbouring molecules, with a Cg2···Cg2^{ii} distance of 3.767 (1) Å and an interplanar distance of 3.452 (1) Å resulting in a slippage of 1.508 Å (Cg2 is the centroid of the C1/C2/C7/O1/C8 furan ring).

S2. Experimental

77% 3-chloroperoxybenzoic acid (269 mg, 1.2 mmol) was added in small portions to a stirred solution of 5-bromo-3-cyclohexylsulfanyl-2,7-dimethyl-1-benzofuran (373 mg, 1.1 mmol) in dichloromethane (40 mL) at 273 K. After being stirred at room temperature for 3h, 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 75%, m.p. 415–416 K; R_f = 0.61 (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 acetone 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, 1.00 Å for methine, 0.99 Å for methylene and 0.98 Å for methyl H atoms, respectively. U_{iso}(H) =1.2U_{eq}(C) for aryl, methine and methylene, and 1.5U_{eq}(C) for methyl H atoms.

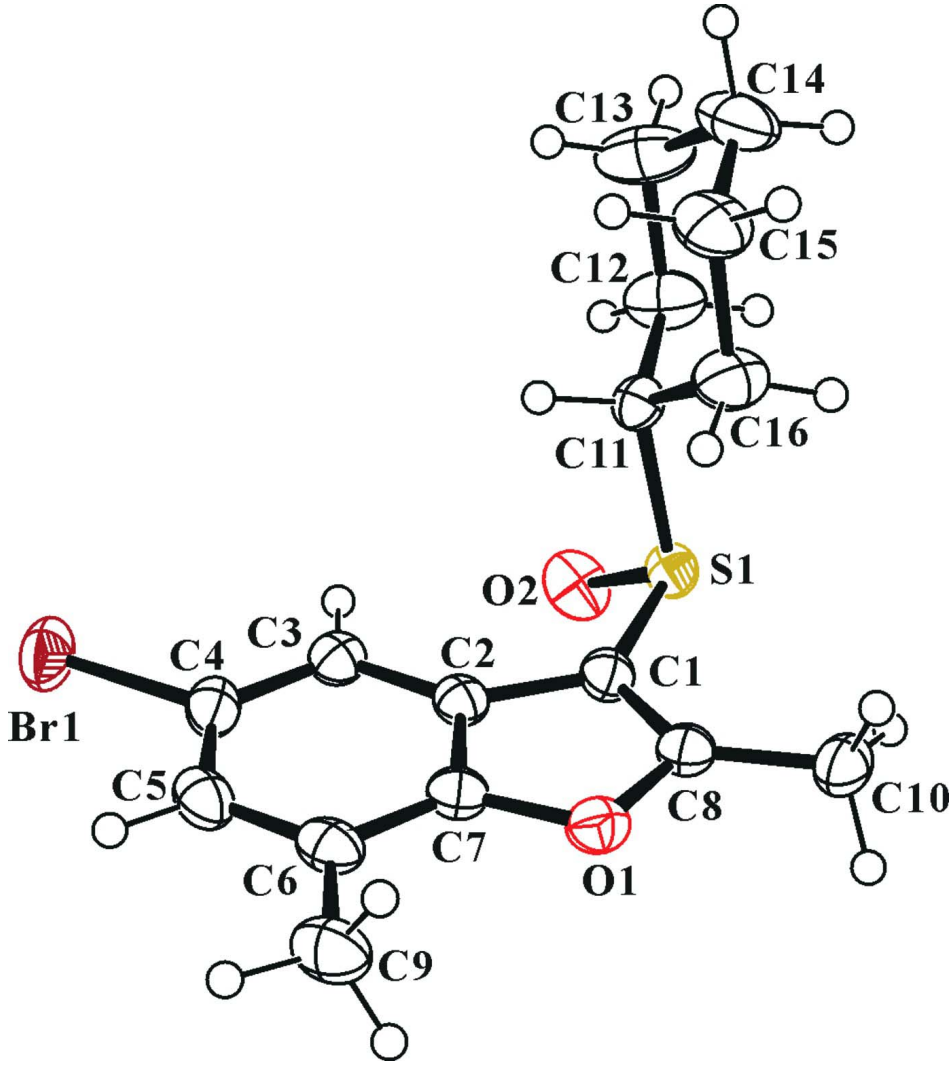

Figure 1

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.

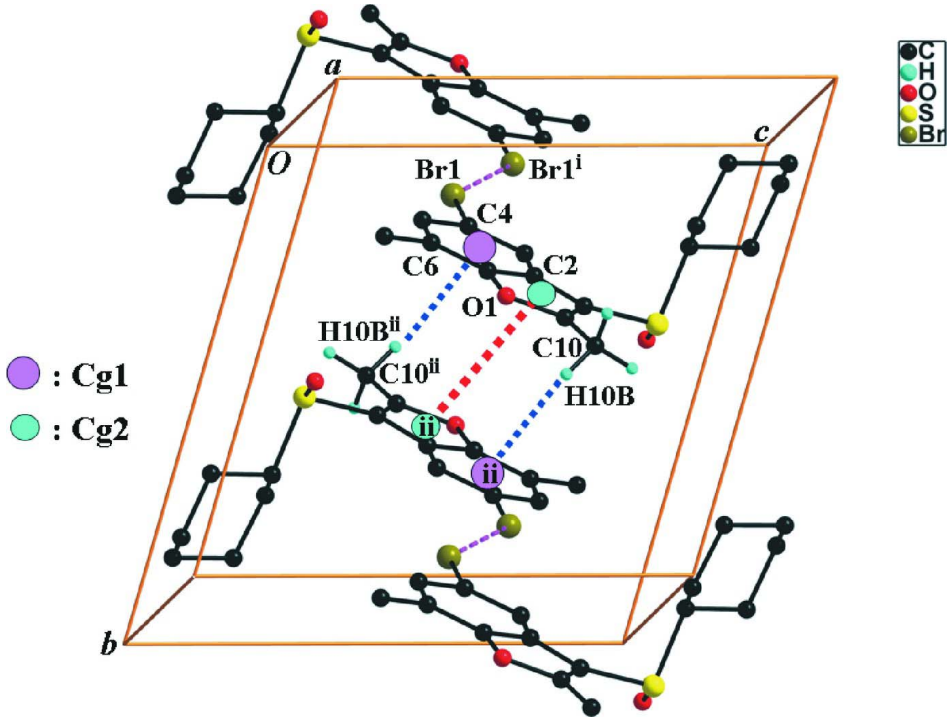

Figure 2

A view of the Br···Br, C—H···π and π···π interactions (dotted lines) in the crystal structure of the title compound. [Symmery codes: (i) -x -1, -*y*, -*z* + 1 ; (ii) -*x* + 1, -*y* + 1, -*z* + 1]

5-Bromo-3-cyclohexylsulfinyl-2,7-dimethyl-1-benzofuran

Crystal data

$C_{16}H_{19}BrO_2S$
M_r = 355.28
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 6.3957 (2) Å
b = 11.2576 (3) Å
c = 12.0384 (3) Å
α = 104.726 (2)°
β = 101.426 (1)°
γ = 106.380 (1)°
V = 769.53 (4) Å^3
Z = 2
F(000) = 364
D_x = 1.533 Mg m^{-3}
Mo *K*α radiation, λ = 0.71073 Å
Cell parameters from 5547 reflections
θ = 3.1–28.1°
μ = 2.81 mm^{-1}
T = 173 K
Block, colourless
0.19 × 0.17 × 0.09 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} = 0.660, T_{max} = 0.746
14554 measured reflections
3887 independent reflections
3165 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 28.6°, θ_{min} = 1.8°
h = −8→8
k = −15→15
l = −15→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.077$
$S = 1.03$
3887 reflections
183 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
$w = 1/[\sigma^2(F_o^2) + (0.0379P)^2 + 0.1985P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ 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	−0.29117 (3)	0.05949 (2)	0.42781 (2)	0.04566 (9)
S1	0.59311 (8)	0.44037 (4)	0.82300 (4)	0.02878 (11)
O1	0.6631 (2)	0.39267 (13)	0.49722 (12)	0.0317 (3)
O2	0.3652 (2)	0.44013 (15)	0.83380 (13)	0.0419 (3)
C1	0.5706 (3)	0.40074 (17)	0.66863 (16)	0.0274 (4)
C2	0.3721 (3)	0.31269 (17)	0.57099 (16)	0.0267 (4)
C3	0.1515 (3)	0.23721 (17)	0.56098 (17)	0.0292 (4)
H3	0.0991	0.2367	0.6295	0.035*
C4	0.0127 (3)	0.16314 (18)	0.44627 (18)	0.0326 (4)
C5	0.0853 (3)	0.16066 (19)	0.34375 (18)	0.0348 (4)
H5	−0.0172	0.1066	0.2670	0.042*
C6	0.3041 (3)	0.23572 (19)	0.35242 (17)	0.0328 (4)
C7	0.4401 (3)	0.31112 (18)	0.46765 (17)	0.0294 (4)
C8	0.7373 (3)	0.44626 (17)	0.62025 (17)	0.0291 (4)
C9	0.3937 (4)	0.2351 (2)	0.24595 (18)	0.0441 (5)
H9A	0.2719	0.1783	0.1722	0.066*
H9B	0.5203	0.2021	0.2532	0.066*
H9C	0.4472	0.3245	0.2430	0.066*
C10	0.9769 (3)	0.53593 (19)	0.67278 (19)	0.0356 (4)
H10A	1.0015	0.5858	0.7568	0.053*
H10B	1.0100	0.5967	0.6276	0.053*
H10C	1.0781	0.4848	0.6688	0.053*
C11	0.6185 (3)	0.28811 (17)	0.84033 (16)	0.0272 (4)
H11	0.4882	0.2124	0.7792	0.033*
C12	0.6042 (5)	0.2884 (2)	0.96458 (19)	0.0467 (5)

H12A	0.4538	0.2901	0.9724	0.056*
H12B	0.7234	0.3675	1.0260	0.056*
C13	0.6374 (6)	0.1651 (3)	0.9844 (2)	0.0619 (7)
H13A	0.6342	0.1671	1.0667	0.074*
H13B	0.5101	0.0867	0.9272	0.074*
C14	0.8601 (5)	0.1549 (2)	0.9674 (2)	0.0578 (7)
H14A	0.9883	0.2295	1.0288	0.069*
H14B	0.8736	0.0728	0.9784	0.069*
C15	0.8734 (4)	0.1555 (2)	0.8440 (2)	0.0482 (6)
H15A	0.7550	0.0757	0.7831	0.058*
H15B	1.0239	0.1537	0.8364	0.058*
C16	0.8397 (4)	0.2765 (2)	0.8201 (2)	0.0447 (5)
H16A	0.9689	0.3559	0.8741	0.054*
H16B	0.8369	0.2704	0.7362	0.054*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02694 (11)	0.03510 (12)	0.06310 (16)	0.00642 (8)	0.00168 (9)	0.01043 (10)
S1	0.0298 (2)	0.0258 (2)	0.0286 (2)	0.01108 (18)	0.00634 (18)	0.00554 (17)
O1	0.0329 (7)	0.0338 (7)	0.0342 (7)	0.0138 (6)	0.0130 (6)	0.0160 (6)
O2	0.0401 (8)	0.0503 (8)	0.0411 (8)	0.0274 (7)	0.0152 (7)	0.0090 (7)
C1	0.0263 (8)	0.0269 (8)	0.0303 (9)	0.0115 (7)	0.0074 (7)	0.0099 (7)
C2	0.0288 (9)	0.0240 (8)	0.0294 (9)	0.0135 (7)	0.0068 (7)	0.0089 (7)
C3	0.0280 (9)	0.0283 (8)	0.0352 (10)	0.0139 (7)	0.0104 (7)	0.0116 (7)
C4	0.0255 (8)	0.0260 (8)	0.0441 (11)	0.0116 (7)	0.0042 (8)	0.0096 (8)
C5	0.0378 (10)	0.0302 (9)	0.0336 (10)	0.0172 (8)	0.0015 (8)	0.0066 (8)
C6	0.0414 (10)	0.0326 (9)	0.0301 (10)	0.0215 (8)	0.0085 (8)	0.0120 (8)
C7	0.0321 (9)	0.0291 (8)	0.0324 (10)	0.0153 (8)	0.0099 (8)	0.0135 (7)
C8	0.0302 (9)	0.0273 (8)	0.0327 (10)	0.0126 (7)	0.0077 (7)	0.0129 (7)
C9	0.0583 (14)	0.0483 (12)	0.0298 (10)	0.0230 (11)	0.0128 (10)	0.0147 (9)
C10	0.0286 (9)	0.0327 (9)	0.0464 (12)	0.0095 (8)	0.0109 (8)	0.0156 (9)
C11	0.0284 (9)	0.0241 (8)	0.0285 (9)	0.0093 (7)	0.0093 (7)	0.0070 (7)
C12	0.0718 (16)	0.0448 (12)	0.0364 (11)	0.0302 (12)	0.0247 (11)	0.0171 (10)
C13	0.107 (2)	0.0588 (15)	0.0489 (14)	0.0436 (16)	0.0409 (15)	0.0349 (13)
C14	0.0807 (19)	0.0438 (12)	0.0458 (13)	0.0323 (13)	−0.0038 (12)	0.0149 (10)
C15	0.0461 (12)	0.0492 (13)	0.0619 (15)	0.0308 (11)	0.0163 (11)	0.0229 (11)
C16	0.0388 (11)	0.0510 (12)	0.0653 (15)	0.0273 (10)	0.0260 (11)	0.0325 (11)

Geometric parameters (Å, º)

Br1—C4	1.8987 (19)	C9—H9C	0.9800
Br1—Br1^{i}	3.5994 (5)	C10—H10A	0.9800
S1—O2	1.4885 (14)	C10—H10B	0.9800
S1—C1	1.7634 (19)	C10—H10C	0.9800
S1—C11	1.8259 (18)	C11—C12	1.516 (3)
O1—C8	1.374 (2)	C11—C16	1.517 (3)
O1—C7	1.378 (2)	C11—H11	1.0000

C1—C8	1.352 (3)	C12—C13	1.530 (3)
C1—C2	1.443 (2)	C12—H12A	0.9900
C2—C3	1.390 (3)	C12—H12B	0.9900
C2—C7	1.395 (3)	C13—C14	1.510 (4)
C3—C4	1.379 (3)	C13—H13A	0.9900
C3—H3	0.9500	C13—H13B	0.9900
C4—C5	1.399 (3)	C14—C15	1.507 (3)
C5—C6	1.383 (3)	C14—H14A	0.9900
C5—H5	0.9500	C14—H14B	0.9900
C6—C7	1.381 (3)	C15—C16	1.526 (3)
C6—C9	1.503 (3)	C15—H15A	0.9900
C8—C10	1.479 (3)	C15—H15B	0.9900
C9—H9A	0.9800	C16—H16A	0.9900
C9—H9B	0.9800	C16—H16B	0.9900

C4—Br1—Br1^{i}	147.22 (6)	H10A—C10—H10C	109.5
O2—S1—C1	106.24 (8)	H10B—C10—H10C	109.5
O2—S1—C11	107.06 (9)	C12—C11—C16	112.33 (17)
C1—S1—C11	97.42 (8)	C12—C11—S1	107.98 (13)
C8—O1—C7	106.57 (14)	C16—C11—S1	109.89 (13)
C8—C1—C2	107.53 (16)	C12—C11—H11	108.9
C8—C1—S1	126.17 (14)	C16—C11—H11	108.9
C2—C1—S1	126.30 (14)	S1—C11—H11	108.9
C3—C2—C7	119.63 (17)	C11—C12—C13	109.28 (18)
C3—C2—C1	135.63 (18)	C11—C12—H12A	109.8
C7—C2—C1	104.75 (16)	C13—C12—H12A	109.8
C4—C3—C2	116.39 (18)	C11—C12—H12B	109.8
C4—C3—H3	121.8	C13—C12—H12B	109.8
C2—C3—H3	121.8	H12A—C12—H12B	108.3
C3—C4—C5	123.19 (18)	C14—C13—C12	111.7 (2)
C3—C4—Br1	117.98 (15)	C14—C13—H13A	109.3
C5—C4—Br1	118.83 (14)	C12—C13—H13A	109.3
C6—C5—C4	120.99 (18)	C14—C13—H13B	109.3
C6—C5—H5	119.5	C12—C13—H13B	109.3
C4—C5—H5	119.5	H13A—C13—H13B	107.9
C7—C6—C5	115.22 (18)	C15—C14—C13	110.68 (19)
C7—C6—C9	121.3 (2)	C15—C14—H14A	109.5
C5—C6—C9	123.49 (19)	C13—C14—H14A	109.5
O1—C7—C6	125.06 (17)	C15—C14—H14B	109.5
O1—C7—C2	110.37 (16)	C13—C14—H14B	109.5
C6—C7—C2	124.55 (18)	H14A—C14—H14B	108.1
C1—C8—O1	110.78 (16)	C14—C15—C16	111.53 (19)
C1—C8—C10	133.19 (18)	C14—C15—H15A	109.3
O1—C8—C10	116.00 (16)	C16—C15—H15A	109.3
C6—C9—H9A	109.5	C14—C15—H15B	109.3
C6—C9—H9B	109.5	C16—C15—H15B	109.3
H9A—C9—H9B	109.5	H15A—C15—H15B	108.0
C6—C9—H9C	109.5	C11—C16—C15	110.72 (18)

H9A—C9—H9C	109.5	C11—C16—H16A	109.5
H9B—C9—H9C	109.5	C15—C16—H16A	109.5
C8—C10—H10A	109.5	C11—C16—H16B	109.5
C8—C10—H10B	109.5	C15—C16—H16B	109.5
H10A—C10—H10B	109.5	H16A—C16—H16B	108.1
C8—C10—H10C	109.5		

O2—S1—C1—C8	146.51 (16)	C9—C6—C7—C2	−177.25 (17)
C11—S1—C1—C8	−103.24 (17)	C3—C2—C7—O1	179.67 (15)
O2—S1—C1—C2	−34.29 (17)	C1—C2—C7—O1	−0.58 (19)
C11—S1—C1—C2	75.95 (16)	C3—C2—C7—C6	−1.8 (3)
C8—C1—C2—C3	−179.44 (19)	C1—C2—C7—C6	177.92 (17)
S1—C1—C2—C3	1.2 (3)	C2—C1—C8—O1	−0.9 (2)
C8—C1—C2—C7	0.88 (19)	S1—C1—C8—O1	178.45 (12)
S1—C1—C2—C7	−178.44 (13)	C2—C1—C8—C10	−178.65 (19)
C7—C2—C3—C4	0.6 (2)	S1—C1—C8—C10	0.7 (3)
C1—C2—C3—C4	−179.02 (18)	C7—O1—C8—C1	0.51 (19)
C2—C3—C4—C5	0.7 (3)	C7—O1—C8—C10	178.71 (15)
C2—C3—C4—Br1	−179.46 (12)	O2—S1—C11—C12	−61.81 (16)
Br1^{i}—Br1—C4—C3	−6.1 (2)	C1—S1—C11—C12	−171.38 (15)
Br1^{i}—Br1—C4—C5	173.74 (9)	O2—S1—C11—C16	175.35 (14)
C3—C4—C5—C6	−1.0 (3)	C1—S1—C11—C16	65.79 (15)
Br1—C4—C5—C6	179.16 (14)	C16—C11—C12—C13	−55.8 (3)
C4—C5—C6—C7	−0.1 (3)	S1—C11—C12—C13	−177.09 (19)
C4—C5—C6—C9	178.64 (18)	C11—C12—C13—C14	56.7 (3)
C8—O1—C7—C6	−178.41 (17)	C12—C13—C14—C15	−57.4 (3)
C8—O1—C7—C2	0.08 (19)	C13—C14—C15—C16	55.9 (3)
C5—C6—C7—O1	179.78 (16)	C12—C11—C16—C15	55.2 (2)
C9—C6—C7—O1	1.0 (3)	S1—C11—C16—C15	175.44 (16)
C5—C6—C7—C2	1.5 (3)	C14—C15—C16—C11	−54.6 (3)

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

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the phenyl ring of the benzofuran.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10*B*···*Cg*1^{ii}	0.98	2.97	3.717 (2)	134

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