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3-(3-Chlorophenylsulfonyl)-2,5-dimethyl-1-benzofuran

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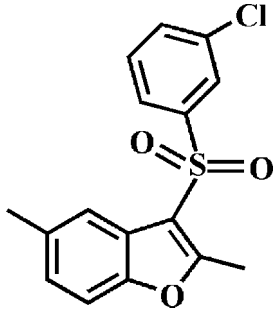
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.110; data-to-parameter ratio = 18.9.

In the title molecule, $C_{16}H_{13}ClO_3S$, the 3-chlorophenyl ring makes a dihedral angle of 76.30 (5)° with the mean plane of the benzofuran fragment. In the crystal, pairs of intermolecular C—H$\cdots\pi$ interactions link the molecules into inversion dimers.

Related literature

For the pharmacological activity of benzofuran compounds, see: Aslam *et al.* (2009); 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 3-(4-chlorophenylsulfonyl)-2-methyl-1-benzofuran derivatives, see: Choi *et al.* (2010, 2011).



Experimental

Crystal data

$C_{16}H_{13}ClO_3S$ M_r = 320.77
Triclinic, $P\overline{1}$
a = 8.1000 (4) Å
b = 8.8622 (5) Å
c = 10.6898 (6) Å
α = 84.023 (3)°
β = 81.514 (3)°
γ = 72.378 (3)°
V = 721.90 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.42 mm^{-1}
T = 173 K
0.28 × 0.27 × 0.19 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.579, T_{max} = 0.746
13538 measured reflections
3622 independent reflections
2951 reflections with $I > 2\sigma(I)$
R_{int} = 0.051

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.110
S = 1.05
3622 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

Cg is the centroid of the C2–C7 benzene ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C9—H9*C*$\cdots$*Cg*^{i}	0.98	2.73	3.663 (2)	159

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

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: QM2026).

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3-(3-Chlorophenylsulfonyl)-2,5-dimethyl-1-benzofuran

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S1. Comment

Recently, many compounds containing a benzofuran ring system have drawn much attention owing to their valuable pharmacological properties such as antibacterial and antifungal, antitumor and antiviral, and antimicrobial activities (Aslam *et al.*, 2009, Galal *et al.*, 2009, Khan *et al.*, 2005). These benzofuran derivatives 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-(4-chlorophenylsulfonyl)-2-methyl-1-benzofuran analogues (Choi *et al.*, 2010, 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 0.004 (1) Å from the least-squares plane defined by the nine constituent atoms. The dihedral angle formed by the 3-chlorophenyl ring and the mean plane of the benzofuran fragment is 76.30 (5)° . The crystal packing (Fig.2) is stabilized by intermolecular C—H···π interactions between a methyl H atom and the benzene ring (Table 1; C9—H9C···Cg^{i}, Cg is the centroid of the C2–C7 benzene ring).

S2. Experimental

77% 3-chloroperoxybenzoic acid (514 mg, 2.3 mmol) was added in small portions to a stirred solution of 3-(3-chlorophenylsulfanyl)-2,5-dimethyl-1-benzofuran (317 mg, 1.1 mmol) in dichloromethane (30 mL) at 273 K. After being stirred at room temperature for 4h, 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. 396–397 K; Rf = 0.75 (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 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 and 0.99 Å for methyl H atoms. U_{iso}(H) = 1.2U_{eq}(C) for aryl and 1.5U_{eq}(C) for methyl H atoms.

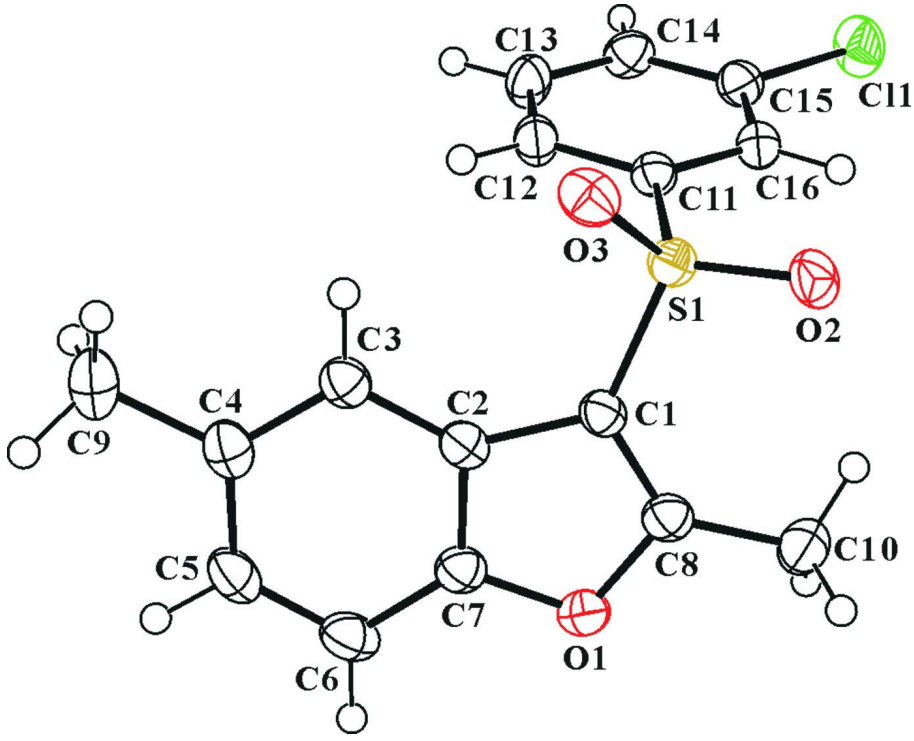

Figure 1

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 radius.

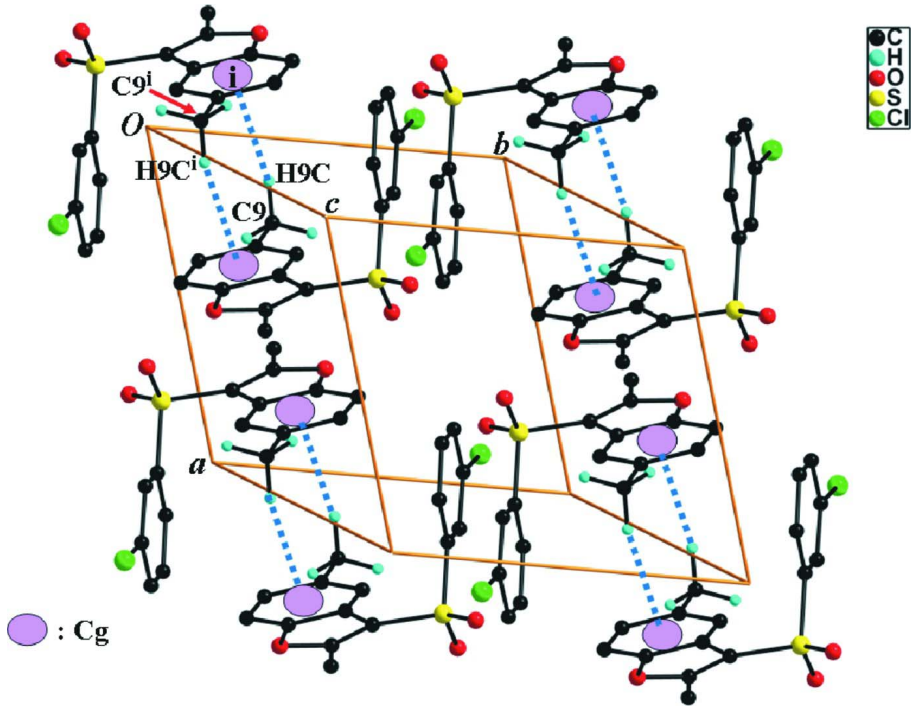

Figure 2

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

3-(3-Chlorophenylsulfonyl)-2,5-dimethyl-1-benzofuran

Crystal data

$C_{16}H_{13}ClO_3S$
M_r = 320.77
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 8.1000 (4) Å
b = 8.8622 (5) Å
c = 10.6898 (6) Å
α = 84.023 (3)°
β = 81.514 (3)°
γ = 72.378 (3)°
V = 721.90 (7) Å^3
Z = 2
F(000) = 332
D_x = 1.476 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5186 reflections
θ = 2.4–27.9°
μ = 0.42 mm^{-1}
T = 173 K
Block, colourless
0.28 × 0.27 × 0.19 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} = 0.579, T_{max} = 0.746
13538 measured reflections
3622 independent reflections
2951 reflections with $I > 2\sigma(I)$
R_{int} = 0.051
θ_{max} = 28.4°, θ_{min} = 1.9°
h = −10→10
k = −11→11
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.110$
$S = 1.05$
3622 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
$w = 1/[\sigma^2(F_o^2) + (0.0497P)^2 + 0.243P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.40$ 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}
S1	0.33167 (5)	0.43504 (5)	0.29715 (4)	0.02475 (13)
Cl1	−0.08568 (7)	0.78553 (6)	−0.04210 (5)	0.04248 (16)
O1	0.49244 (16)	−0.00637 (14)	0.20581 (12)	0.0310 (3)
O2	0.45023 (15)	0.49763 (14)	0.20924 (13)	0.0328 (3)
O3	0.31247 (17)	0.46293 (15)	0.42888 (12)	0.0324 (3)
C1	0.3859 (2)	0.23236 (19)	0.28520 (16)	0.0239 (3)
C2	0.3302 (2)	0.12036 (19)	0.37751 (16)	0.0242 (3)
C3	0.2342 (2)	0.1260 (2)	0.49708 (16)	0.0272 (4)
H3	0.1872	0.2233	0.5372	0.033*
C4	0.2087 (2)	−0.0133 (2)	0.55658 (17)	0.0298 (4)
C5	0.2811 (2)	−0.1561 (2)	0.49675 (19)	0.0342 (4)
H5	0.2626	−0.2505	0.5389	0.041*
C6	0.3782 (2)	−0.1647 (2)	0.3793 (2)	0.0341 (4)
H6	0.4273	−0.2621	0.3395	0.041*
C7	0.3998 (2)	−0.0244 (2)	0.32300 (17)	0.0274 (4)
C8	0.4815 (2)	0.1509 (2)	0.18512 (17)	0.0273 (4)
C9	0.1031 (3)	−0.0119 (3)	0.68477 (19)	0.0404 (5)
H9A	0.1140	0.0730	0.7319	0.061*
H9B	0.1462	−0.1143	0.7316	0.061*
H9C	−0.0198	0.0063	0.6744	0.061*
C10	0.5727 (2)	0.1939 (2)	0.06278 (18)	0.0367 (4)
H10A	0.5343	0.3093	0.0459	0.055*
H10B	0.5455	0.1426	−0.0052	0.055*
H10C	0.6989	0.1584	0.0665	0.055*
C11	0.1232 (2)	0.51080 (19)	0.24554 (15)	0.0241 (3)

C12	−0.0193 (2)	0.4771 (2)	0.31850 (17)	0.0287 (4)
H12	−0.0056	0.4149	0.3963	0.034*
C13	−0.1810 (2)	0.5353 (2)	0.27665 (18)	0.0326 (4)
H13	−0.2789	0.5109	0.3250	0.039*
C14	−0.2017 (2)	0.6286 (2)	0.16502 (17)	0.0313 (4)
H14	−0.3133	0.6683	0.1362	0.038*
C15	−0.0585 (2)	0.6636 (2)	0.09564 (17)	0.0290 (4)
C16	0.1054 (2)	0.6038 (2)	0.13328 (16)	0.0277 (4)
H16	0.2037	0.6259	0.0835	0.033*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0225 (2)	0.0215 (2)	0.0316 (2)	−0.00858 (15)	−0.00493 (16)	0.00078 (16)
Cl1	0.0428 (3)	0.0451 (3)	0.0349 (3)	−0.0092 (2)	−0.0093 (2)	0.0141 (2)
O1	0.0274 (6)	0.0253 (6)	0.0376 (7)	−0.0053 (5)	0.0013 (5)	−0.0050 (5)
O2	0.0245 (6)	0.0294 (7)	0.0460 (8)	−0.0130 (5)	−0.0032 (5)	0.0051 (5)
O3	0.0383 (7)	0.0286 (6)	0.0335 (7)	−0.0114 (5)	−0.0106 (5)	−0.0027 (5)
C1	0.0210 (7)	0.0221 (7)	0.0294 (8)	−0.0071 (6)	−0.0048 (6)	0.0008 (6)
C2	0.0198 (7)	0.0225 (8)	0.0315 (9)	−0.0071 (6)	−0.0072 (6)	0.0010 (6)
C3	0.0259 (8)	0.0260 (8)	0.0300 (9)	−0.0075 (6)	−0.0059 (7)	0.0003 (7)
C4	0.0257 (8)	0.0330 (9)	0.0325 (9)	−0.0115 (7)	−0.0093 (7)	0.0067 (7)
C5	0.0314 (9)	0.0254 (9)	0.0469 (11)	−0.0114 (7)	−0.0093 (8)	0.0083 (8)
C6	0.0310 (9)	0.0222 (8)	0.0495 (12)	−0.0075 (7)	−0.0062 (8)	−0.0024 (8)
C7	0.0219 (8)	0.0256 (8)	0.0343 (9)	−0.0062 (6)	−0.0040 (7)	−0.0008 (7)
C8	0.0218 (8)	0.0256 (8)	0.0339 (9)	−0.0059 (6)	−0.0053 (7)	0.0001 (7)
C9	0.0420 (11)	0.0465 (11)	0.0343 (10)	−0.0199 (9)	−0.0036 (8)	0.0096 (9)
C10	0.0323 (9)	0.0402 (10)	0.0331 (10)	−0.0079 (8)	0.0025 (8)	0.0003 (8)
C11	0.0222 (8)	0.0223 (8)	0.0273 (8)	−0.0061 (6)	−0.0024 (6)	−0.0010 (6)
C12	0.0264 (8)	0.0293 (9)	0.0288 (9)	−0.0083 (7)	−0.0019 (7)	0.0047 (7)
C13	0.0237 (8)	0.0375 (10)	0.0349 (10)	−0.0095 (7)	0.0001 (7)	0.0023 (8)
C14	0.0241 (8)	0.0338 (9)	0.0330 (9)	−0.0034 (7)	−0.0054 (7)	−0.0011 (7)
C15	0.0307 (9)	0.0263 (8)	0.0270 (9)	−0.0046 (7)	−0.0045 (7)	0.0016 (7)
C16	0.0264 (8)	0.0257 (8)	0.0298 (9)	−0.0079 (7)	−0.0008 (7)	0.0012 (7)

Geometric parameters (Å, º)

S1—O3	1.4328 (13)	C6—H6	0.9500
S1—O2	1.4355 (12)	C8—C10	1.478 (2)
S1—C1	1.7289 (17)	C9—H9A	0.9800
S1—C11	1.7659 (17)	C9—H9B	0.9800
Cl1—C15	1.7334 (18)	C9—H9C	0.9800
O1—C8	1.366 (2)	C10—H10A	0.9800
O1—C7	1.383 (2)	C10—H10B	0.9800
C1—C8	1.357 (2)	C10—H10C	0.9800
C1—C2	1.449 (2)	C11—C16	1.383 (2)
C2—C7	1.387 (2)	C11—C12	1.387 (2)
C2—C3	1.392 (2)	C12—C13	1.377 (3)

C3—C4	1.386 (2)	C12—H12	0.9500
C3—H3	0.9500	C13—C14	1.380 (3)
C4—C5	1.401 (3)	C13—H13	0.9500
C4—C9	1.504 (3)	C14—C15	1.381 (2)
C5—C6	1.376 (3)	C14—H14	0.9500
C5—H5	0.9500	C15—C16	1.376 (2)
C6—C7	1.373 (3)	C16—H16	0.9500

O3—S1—O2	119.46 (8)	C4—C9—H9A	109.5
O3—S1—C1	107.56 (8)	C4—C9—H9B	109.5
O2—S1—C1	109.13 (8)	H9A—C9—H9B	109.5
O3—S1—C11	107.49 (8)	C4—C9—H9C	109.5
O2—S1—C11	107.47 (8)	H9A—C9—H9C	109.5
C1—S1—C11	104.80 (8)	H9B—C9—H9C	109.5
C8—O1—C7	106.97 (13)	C8—C10—H10A	109.5
C8—C1—C2	107.83 (15)	C8—C10—H10B	109.5
C8—C1—S1	126.08 (13)	H10A—C10—H10B	109.5
C2—C1—S1	126.02 (12)	C8—C10—H10C	109.5
C7—C2—C3	119.05 (16)	H10A—C10—H10C	109.5
C7—C2—C1	104.37 (15)	H10B—C10—H10C	109.5
C3—C2—C1	136.58 (16)	C16—C11—C12	121.36 (16)
C4—C3—C2	118.71 (16)	C16—C11—S1	119.09 (12)
C4—C3—H3	120.6	C12—C11—S1	119.55 (13)
C2—C3—H3	120.6	C13—C12—C11	119.07 (16)
C3—C4—C5	119.84 (16)	C13—C12—H12	120.5
C3—C4—C9	120.42 (17)	C11—C12—H12	120.5
C5—C4—C9	119.74 (17)	C12—C13—C14	120.52 (16)
C6—C5—C4	122.43 (17)	C12—C13—H13	119.7
C6—C5—H5	118.8	C14—C13—H13	119.7
C4—C5—H5	118.8	C13—C14—C15	119.29 (17)
C7—C6—C5	116.06 (17)	C13—C14—H14	120.4
C7—C6—H6	122.0	C15—C14—H14	120.4
C5—C6—H6	122.0	C16—C15—C14	121.53 (16)
C6—C7—O1	125.54 (16)	C16—C15—Cl1	119.16 (14)
C6—C7—C2	123.90 (16)	C14—C15—Cl1	119.31 (14)
O1—C7—C2	110.56 (15)	C15—C16—C11	118.19 (16)
C1—C8—O1	110.26 (15)	C15—C16—H16	120.9
C1—C8—C10	134.82 (17)	C11—C16—H16	120.9
O1—C8—C10	114.92 (15)		

O3—S1—C1—C8	−151.41 (16)	C1—C2—C7—O1	0.08 (19)
O2—S1—C1—C8	−20.43 (19)	C2—C1—C8—O1	−0.2 (2)
C11—S1—C1—C8	94.41 (17)	S1—C1—C8—O1	−177.39 (12)
O3—S1—C1—C2	31.89 (17)	C2—C1—C8—C10	179.7 (2)
O2—S1—C1—C2	162.86 (14)	S1—C1—C8—C10	2.5 (3)
C11—S1—C1—C2	−82.29 (16)	C7—O1—C8—C1	0.2 (2)
C8—C1—C2—C7	0.06 (19)	C7—O1—C8—C10	−179.65 (15)
S1—C1—C2—C7	177.27 (13)	O3—S1—C11—C16	133.45 (14)

C8—C1—C2—C3	179.0 (2)	O2—S1—C11—C16	3.68 (16)
S1—C1—C2—C3	−3.8 (3)	C1—S1—C11—C16	−112.32 (14)
C7—C2—C3—C4	−1.3 (3)	O3—S1—C11—C12	−46.03 (16)
C1—C2—C3—C4	179.93 (18)	O2—S1—C11—C12	−175.80 (14)
C2—C3—C4—C5	0.9 (3)	C1—S1—C11—C12	68.19 (16)
C2—C3—C4—C9	−178.90 (17)	C16—C11—C12—C13	1.3 (3)
C3—C4—C5—C6	−0.1 (3)	S1—C11—C12—C13	−179.25 (14)
C9—C4—C5—C6	179.68 (18)	C11—C12—C13—C14	−1.4 (3)
C4—C5—C6—C7	−0.3 (3)	C12—C13—C14—C15	−0.2 (3)
C5—C6—C7—O1	179.85 (17)	C13—C14—C15—C16	1.9 (3)
C5—C6—C7—C2	−0.1 (3)	C13—C14—C15—Cl1	−178.12 (15)
C8—O1—C7—C6	179.81 (18)	C14—C15—C16—C11	−2.0 (3)
C8—O1—C7—C2	−0.20 (19)	Cl1—C15—C16—C11	178.02 (13)
C3—C2—C7—C6	0.9 (3)	C12—C11—C16—C15	0.4 (3)
C1—C2—C7—C6	−179.93 (17)	S1—C11—C16—C15	−179.09 (13)
C3—C2—C7—O1	−179.07 (15)		

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C2–C7 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9*C*···*Cg*^{i}	0.98	2.73	3.663 (2)	159

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