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5-Chloro-3-cyclopentylsulfinyl-2-methyl-1-benzofuran

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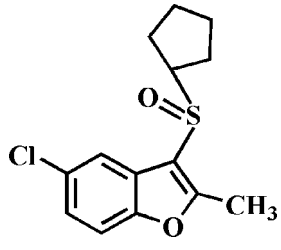
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.105; data-to-parameter ratio = 18.3.

In the title compound, $C_{14}H_{15}ClO_2S$, the cyclopentyl ring adopts an envelope conformation. In the crystal, molecules are linked through weak intermolecular C—H···O hydrogen bonds. The crystal structure also exhibits a slipped π–π interaction between the furan and benzene rings of neighbouring molecules [centroid–centroid distance = 3.784 (3) Å, interplanar distance = 3.199 (3) Å and slippage = 2.021 (3) Å].

Related literature

For the biological 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 a structural study of the related compound, 5-bromo-3-cyclopentylsulfinyl-2-methyl-1-benzofuran, see: Seo *et al.* (2011).



Experimental

Crystal data

$C_{14}H_{15}ClO_2S$ M_r = 282.77
Triclinic, $P\overline{1}$
a = 6.3337 (3) Å
b = 8.9449 (4) Å
c = 12.1157 (5) Å
α = 73.614 (2)°
β = 78.110 (2)°
γ = 88.087 (2)°
V = 644.17 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.45 mm^{-1}
T = 173 K
0.35 × 0.26 × 0.20 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.858, T_{max} = 0.916
11628 measured reflections
2995 independent reflections
2514 reflections with $I > 2\sigma(I)$
R_{int} = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.105
S = 1.05
2995 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O2^{i}	0.95	2.61	3.349 (2)	135

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

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* (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: ZL2373).

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supporting information

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5-Chloro-3-cyclopentylsulfinyl-2-methyl-1-benzofuran

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S1. Comment

Recently, many compounds involving a benzofuran ring system have drawn much attention due to their inetresting 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 part of our ongoing project of the substituent effect on the solid state structures of 3-cyclopentylsulfinyl-5-halo-2-methyl-1-benzofuran analogues (Seo *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 0.009 (1) Å from the least-squares plane defined by the nine constituent atoms. The cyclopentyl ring is in the envelope form. The crystal packing (Fig. 2) is stabilized by weak intermolecular C—H···O hydrogen bonds between a benzene H atom and the O atom of the sulfinyl group (Table 1; C5—H5···O2^{i}). The crystal packing (Fig. 2) is further stabilized by a weak slipped π–π interaction between the furan and benzene rings of neighbouring molecules, with a Cg1···Cg2^{ii} distance of 3.784 (3) Å and an interplanar distance of 3.199 (3) Å resulting in a slippage of 2.021 (3) Å (Cg1 and Cg2 are the centroids of the C1/C2/C7/O1/C8 furan ring and the C2–C7 benzene ring, respectively).

S2. Experimental

77% 3-chloroperoxybenzoic acid (269 mg, 1.2 mmol) was added in small portions to a stirred solution of 5-chloro-3-cyclopentylsulfanyl-2-methy1-benzofuran (293 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 anhydrous magnesium sulfate, filtered and concentrated at reduced pressure. The residue was purified by column chromatography (hexane–ethyl acetate, 1:1 v/v) to afford the title compound as a colorless solid [yield 74%, m.p. 386–387 K; R_f = 0.48 (hexane–ethyl acetate, 1:1 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, 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, methylene, and 1.5U_{eq}(C) for methyl H atoms.

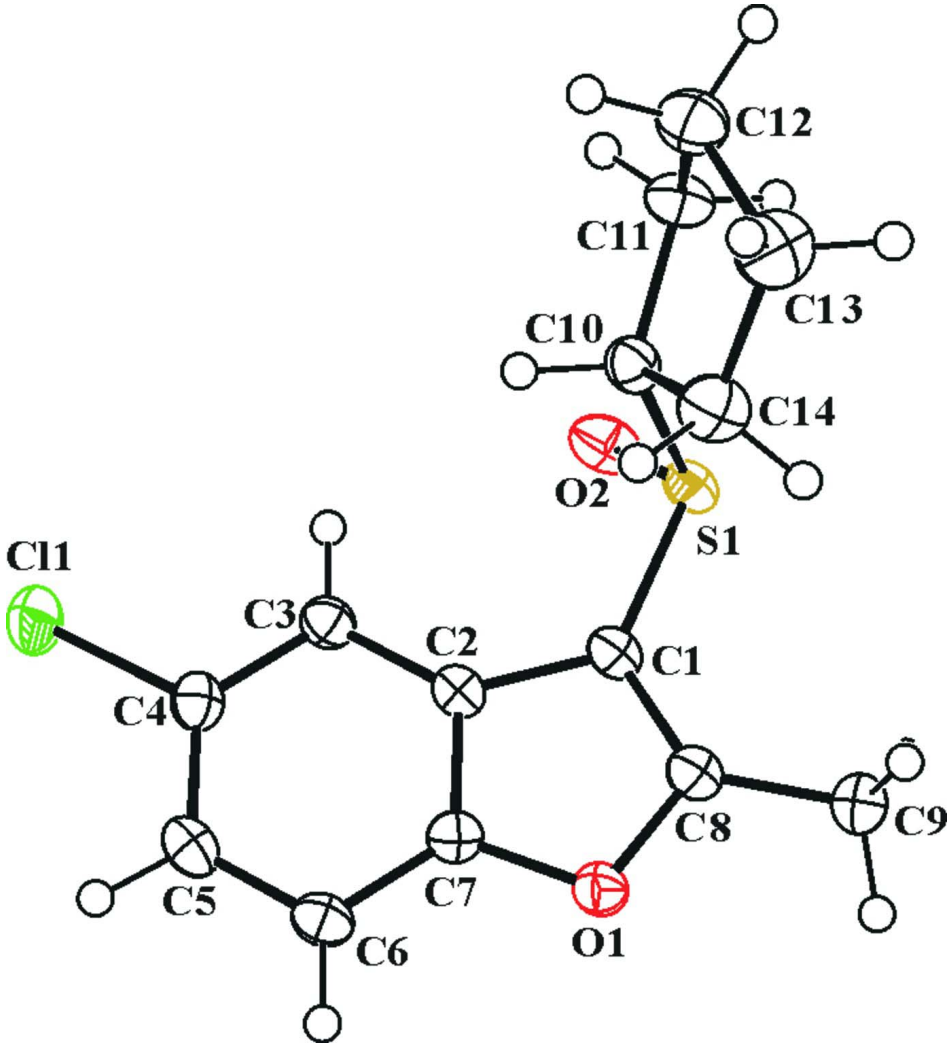

Figure 1

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 a small spheres of arbitrary radius.

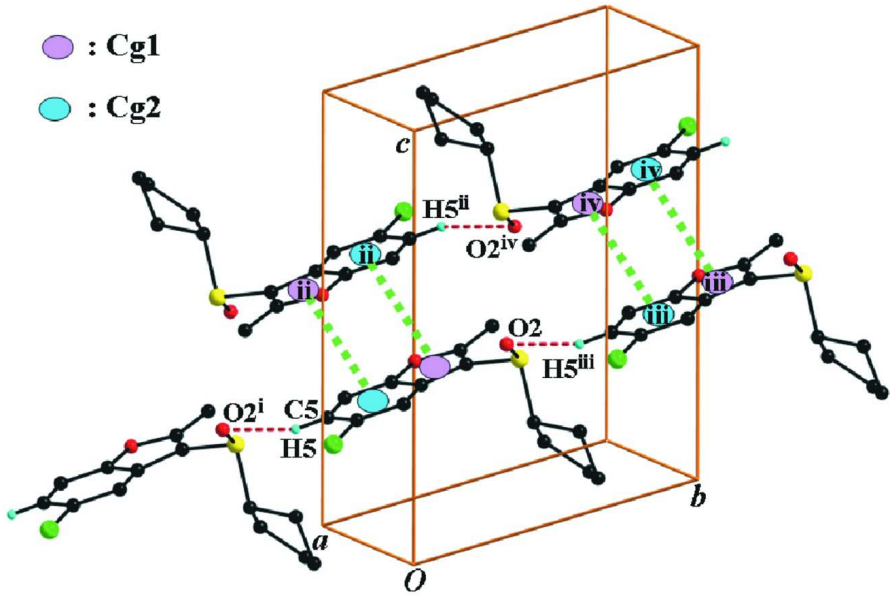

Figure 2

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

5-Chloro-3-cyclopentylsulfinyl-2-methyl-1-benzofuran

Crystal data

$C_{14}H_{15}ClO_2S$
M_r = 282.77
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 6.3337 (3) Å
b = 8.9449 (4) Å
c = 12.1157 (5) Å
α = 73.614 (2)°
β = 78.110 (2)°
γ = 88.087 (2)°
V = 644.17 (5) Å^3

Z = 2
F(000) = 296
D_x = 1.458 Mg m^{-3}
Mo *K*α radiation, λ = 0.71073 Å
Cell parameters from 4102 reflections
θ = 2.4–27.6°
μ = 0.45 mm^{-1}
T = 173 K
Block, colourless
0.35 × 0.26 × 0.20 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} = 0.858, T_{max} = 0.916

11628 measured reflections
2995 independent reflections
2514 reflections with $I > 2\sigma(I)$
R_{int} = 0.055
θ_{max} = 27.7°, θ_{min} = 1.8°
h = −8→8
k = −11→11
l = −15→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.105$
$S = 1.05$
2995 reflections
164 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
$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2 + 0.2468P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ 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}
Cl1	1.11290 (7)	0.07397 (6)	0.16222 (5)	0.03628 (15)
S1	0.45480 (7)	0.51787 (5)	0.33115 (4)	0.02483 (14)
O1	0.24300 (19)	0.08162 (14)	0.43094 (11)	0.0254 (3)
O2	0.6818 (2)	0.54497 (15)	0.33897 (13)	0.0345 (3)
C1	0.4123 (3)	0.3158 (2)	0.36271 (16)	0.0238 (4)
C2	0.5584 (3)	0.2048 (2)	0.32358 (15)	0.0216 (4)
C3	0.7684 (3)	0.2107 (2)	0.25933 (16)	0.0238 (4)
H3	0.8512	0.3054	0.2287	0.029*
C4	0.8508 (3)	0.0724 (2)	0.24220 (16)	0.0247 (4)
C5	0.7343 (3)	−0.0685 (2)	0.28622 (17)	0.0267 (4)
H5	0.7976	−0.1606	0.2714	0.032*
C6	0.5269 (3)	−0.0747 (2)	0.35141 (17)	0.0263 (4)
H6	0.4450	−0.1697	0.3833	0.032*
C7	0.4445 (3)	0.0636 (2)	0.36806 (15)	0.0224 (4)
C8	0.2289 (3)	0.2361 (2)	0.42738 (16)	0.0246 (4)
C9	0.0257 (3)	0.2831 (2)	0.49225 (18)	0.0313 (4)
H9A	0.0574	0.3630	0.5281	0.047*
H9B	−0.0443	0.1921	0.5539	0.047*
H9C	−0.0707	0.3252	0.4378	0.047*
C10	0.4471 (3)	0.5660 (2)	0.17647 (16)	0.0252 (4)
H10	0.5579	0.5056	0.1366	0.030*
C11	0.4876 (3)	0.7408 (2)	0.11699 (17)	0.0305 (4)
H11A	0.4244	0.8030	0.1710	0.037*
H11B	0.6440	0.7665	0.0894	0.037*
C12	0.3739 (3)	0.7690 (2)	0.01427 (18)	0.0343 (4)

H12A	0.3433	0.8808	−0.0155	0.041*
H12B	0.4619	0.7336	−0.0508	0.041*
C13	0.1660 (3)	0.6720 (3)	0.0669 (2)	0.0414 (5)
H13A	0.1129	0.6397	0.0055	0.050*
H13B	0.0528	0.7324	0.1035	0.050*
C14	0.2223 (3)	0.5299 (2)	0.15928 (19)	0.0335 (4)
H14A	0.2250	0.4354	0.1319	0.040*
H14B	0.1150	0.5126	0.2340	0.040*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0254 (2)	0.0385 (3)	0.0432 (3)	0.00585 (19)	−0.0012 (2)	−0.0135 (2)
S1	0.0279 (2)	0.0181 (2)	0.0302 (3)	0.00119 (17)	−0.00814 (18)	−0.00808 (18)
O1	0.0235 (6)	0.0213 (6)	0.0307 (7)	−0.0011 (5)	−0.0035 (5)	−0.0077 (5)
O2	0.0342 (7)	0.0262 (7)	0.0469 (9)	−0.0037 (6)	−0.0199 (6)	−0.0076 (6)
C1	0.0257 (8)	0.0194 (8)	0.0273 (10)	0.0016 (7)	−0.0076 (7)	−0.0069 (7)
C2	0.0236 (8)	0.0205 (8)	0.0230 (9)	0.0018 (6)	−0.0085 (7)	−0.0070 (7)
C3	0.0240 (8)	0.0219 (9)	0.0264 (9)	0.0000 (7)	−0.0070 (7)	−0.0068 (7)
C4	0.0217 (8)	0.0286 (9)	0.0252 (9)	0.0041 (7)	−0.0075 (7)	−0.0084 (8)
C5	0.0318 (9)	0.0228 (9)	0.0298 (10)	0.0052 (7)	−0.0114 (8)	−0.0110 (8)
C6	0.0293 (9)	0.0210 (9)	0.0297 (10)	−0.0024 (7)	−0.0082 (7)	−0.0071 (8)
C7	0.0222 (8)	0.0225 (9)	0.0226 (9)	−0.0009 (6)	−0.0063 (7)	−0.0053 (7)
C8	0.0259 (9)	0.0219 (9)	0.0268 (10)	0.0010 (7)	−0.0071 (7)	−0.0069 (7)
C9	0.0264 (9)	0.0296 (10)	0.0350 (11)	0.0027 (8)	−0.0020 (8)	−0.0079 (9)
C10	0.0249 (9)	0.0228 (9)	0.0289 (10)	0.0010 (7)	−0.0063 (7)	−0.0084 (8)
C11	0.0346 (10)	0.0234 (10)	0.0325 (11)	−0.0034 (8)	−0.0108 (8)	−0.0031 (8)
C12	0.0390 (11)	0.0325 (11)	0.0315 (11)	0.0022 (8)	−0.0120 (9)	−0.0058 (9)
C13	0.0331 (11)	0.0504 (14)	0.0406 (13)	0.0013 (9)	−0.0149 (9)	−0.0074 (11)
C14	0.0294 (10)	0.0325 (11)	0.0404 (12)	−0.0035 (8)	−0.0123 (8)	−0.0087 (9)

Geometric parameters (Å, º)

Cl1—C4	1.7398 (18)	C9—H9A	0.9800
S1—O2	1.4926 (13)	C9—H9B	0.9800
S1—C1	1.7571 (17)	C9—H9C	0.9800
S1—C10	1.8113 (19)	C10—C11	1.532 (2)
O1—C8	1.371 (2)	C10—C14	1.538 (2)
O1—C7	1.375 (2)	C10—H10	1.0000
C1—C8	1.355 (3)	C11—C12	1.520 (3)
C1—C2	1.446 (2)	C11—H11A	0.9900
C2—C7	1.387 (2)	C11—H11B	0.9900
C2—C3	1.390 (2)	C12—C13	1.520 (3)
C3—C4	1.380 (2)	C12—H12A	0.9900
C3—H3	0.9500	C12—H12B	0.9900
C4—C5	1.392 (3)	C13—C14	1.525 (3)
C5—C6	1.380 (3)	C13—H13A	0.9900
C5—H5	0.9500	C13—H13B	0.9900

C6—C7	1.377 (2)	C14—H14A	0.9900
C6—H6	0.9500	C14—H14B	0.9900
C8—C9	1.478 (3)		

O2—S1—C1	107.59 (8)	H9A—C9—H9C	109.5
O2—S1—C10	107.13 (8)	H9B—C9—H9C	109.5
C1—S1—C10	96.85 (8)	C11—C10—C14	105.36 (15)
C8—O1—C7	106.57 (13)	C11—C10—S1	111.26 (12)
C8—C1—C2	107.35 (15)	C14—C10—S1	111.01 (13)
C8—C1—S1	125.01 (14)	C11—C10—H10	109.7
C2—C1—S1	127.63 (14)	C14—C10—H10	109.7
C7—C2—C3	119.67 (16)	S1—C10—H10	109.7
C7—C2—C1	104.65 (15)	C12—C11—C10	102.68 (15)
C3—C2—C1	135.66 (16)	C12—C11—H11A	111.2
C4—C3—C2	116.77 (16)	C10—C11—H11A	111.2
C4—C3—H3	121.6	C12—C11—H11B	111.2
C2—C3—H3	121.6	C10—C11—H11B	111.2
C3—C4—C5	123.11 (17)	H11A—C11—H11B	109.1
C3—C4—Cl1	118.46 (14)	C13—C12—C11	103.48 (17)
C5—C4—Cl1	118.43 (14)	C13—C12—H12A	111.1
C6—C5—C4	120.05 (17)	C11—C12—H12A	111.1
C6—C5—H5	120.0	C13—C12—H12B	111.1
C4—C5—H5	120.0	C11—C12—H12B	111.1
C7—C6—C5	116.80 (16)	H12A—C12—H12B	109.0
C7—C6—H6	121.6	C12—C13—C14	105.84 (16)
C5—C6—H6	121.6	C12—C13—H13A	110.6
O1—C7—C6	125.67 (16)	C14—C13—H13A	110.6
O1—C7—C2	110.74 (15)	C12—C13—H13B	110.6
C6—C7—C2	123.59 (17)	C14—C13—H13B	110.6
C1—C8—O1	110.68 (15)	H13A—C13—H13B	108.7
C1—C8—C9	132.96 (17)	C13—C14—C10	105.96 (16)
O1—C8—C9	116.36 (15)	C13—C14—H14A	110.5
C8—C9—H9A	109.5	C10—C14—H14A	110.5
C8—C9—H9B	109.5	C13—C14—H14B	110.5
H9A—C9—H9B	109.5	C10—C14—H14B	110.5
C8—C9—H9C	109.5	H14A—C14—H14B	108.7

O2—S1—C1—C8	140.45 (16)	C1—C2—C7—O1	−0.31 (19)
C10—S1—C1—C8	−109.10 (16)	C3—C2—C7—C6	−0.7 (3)
O2—S1—C1—C2	−41.40 (18)	C1—C2—C7—C6	−179.57 (16)
C10—S1—C1—C2	69.05 (16)	C2—C1—C8—O1	−1.41 (19)
C8—C1—C2—C7	1.03 (19)	S1—C1—C8—O1	177.05 (12)
S1—C1—C2—C7	−177.38 (13)	C2—C1—C8—C9	178.34 (19)
C8—C1—C2—C3	−177.56 (19)	S1—C1—C8—C9	−3.2 (3)
S1—C1—C2—C3	4.0 (3)	C7—O1—C8—C1	1.22 (18)
C7—C2—C3—C4	0.9 (2)	C7—O1—C8—C9	−178.58 (15)
C1—C2—C3—C4	179.35 (18)	O2—S1—C10—C11	−67.30 (14)
C2—C3—C4—C5	−0.3 (3)	C1—S1—C10—C11	−178.13 (13)

C2—C3—C4—Cl1	179.95 (12)	O2—S1—C10—C14	175.72 (12)
C3—C4—C5—C6	−0.7 (3)	C1—S1—C10—C14	64.89 (14)
Cl1—C4—C5—C6	179.13 (13)	C14—C10—C11—C12	−33.7 (2)
C4—C5—C6—C7	0.9 (3)	S1—C10—C11—C12	−154.09 (13)
C8—O1—C7—C6	178.72 (16)	C10—C11—C12—C13	41.2 (2)
C8—O1—C7—C2	−0.52 (18)	C11—C12—C13—C14	−33.2 (2)
C5—C6—C7—O1	−179.37 (15)	C12—C13—C14—C10	12.1 (2)
C5—C6—C7—C2	−0.2 (3)	C11—C10—C14—C13	13.5 (2)
C3—C2—C7—O1	178.56 (14)	S1—C10—C14—C13	134.01 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O2^{i}	0.95	2.61	3.349 (2)	135

Symmetry code: (i) *x*, *y*−1, *z*.