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5-Chloro-2-phenyl-3-phenylsulfinyl-1-benzofuran

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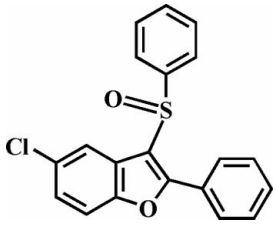
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.003 Å; R factor = 0.034; wR factor = 0.093; data-to-parameter ratio = 15.8.

In the title compound, $C_{20}H_{13}ClO_2S$, the O atom and the phenyl group of the phenylsulfinyl substituent lie on opposite sides of the plane of the benzofuran fragment; the S-bound phenyl ring is nearly perpendicular to this plane [80.87 (5)°]. The phenyl ring in the 2-position is rotated out of the benzofuran plane, making a dihedral angle of 17.43 (7)°. The crystal structure features π–π interactions between the phenyl ring and the furyl ring of a neighbouring benzofuran system [centroid–centroid distance = 3.886 (2) Å].

Related literature

For the crystal structures of similar 2-phenyl-3-phenylsulfinyl-1-benzofuran derivatives, see: Choi *et al.* (2009*a*,*b*). For the pharmacological activity of benzofuran compounds, see: Howlett *et al.* (1999); Twyman & Allsop (1999). For natural products with benzofuran rings, see: Akgul & Anil (2003); von Reuss & König (2004).



Experimental

Crystal data

$C_{20}H_{13}ClO_2S$
M_r = 352.81
Triclinic, $P\overline{1}$
a = 8.2726 (5) Å
b = 9.4111 (5) Å
c = 11.3811 (6) Å
α = 73.360 (1)°
β = 81.630 (1)°
γ = 70.757 (1)°
V = 800.25 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.38 mm^{-1}
T = 273 K
0.24 × 0.15 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1999)
T_{min} = 0.915, T_{max} = 0.963
6930 measured reflections
3428 independent reflections
2843 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.093
S = 1.06
3428 reflections
217 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); 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: NG2614).

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S1. Comment

Molecules containing the benzofuran skeleton have attracted considerable interest in view of their pharmacological properties (Howlett *et al.*, 1999; Twyman & Allsop, 1999) and are well known as natural products (Akgul & Anil, 2003; von Reuss & König, 2004). This work is related to our communications on the synthesis and structures of 2-phenyl-3-phenylsulfiny-1-benzofuran analogues, *viz*. 5-chloro-7-methyl-2-phenyl-3-phenylsulfinyl-1-benzofuran (Choi *et al.*, 2009*a*) and 5-iodo-2-phenyl-3-phenylsulfinyl-1-benzofuran (Choi *et al.*, 2009*b*). Here we report the crystal structure of the title compound (I), 5-chloro-2-phenyl-3-phenylsulfinyl-1-benzofuran (Fig. 1).

The benzofuran unit is essentially planar, with a mean deviation of 0.012 (1) Å from the least-squares plane defined by the nine constituent atoms. The dihedral angle in (I) formed by the plane of the benzofuran ring and the plane of 2-phenyl ring is 17.43 (7)°, and the phenyl ring (C15-C20) with80.87 (5) ° lies toward the benzofuran plane. The crystal packing (Fig. 2) is stabilized by aromatic π–π interactions between the benzene ring and the furan ring from the neighbouring bennzofuran systems. The Cg1···Cg2^{i} distance is 3.886 (2) Å (Cg1 and Cg2 are the centroides of the C2-C7 benzene ring and the C1/C2/C7/O1/C8 furan ring, respectively).

S2. Experimental

The 77% 3-chloroperoxybenzoic acid (157 mg, 0.7 mmol) was added in small portions to a stirred solution of 5-chloro-2-phenyl-3-phenylsulfanyl-1-benzofuran (226 mg, 0.7 mmol) in dichloromethane (30 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 in vacuum. The residue was purified by column chromatography (hexane-ethyl acetate, 2:1 v/v) to afford the title compound as a colorless solid [yield 76%, m.p. 421-422 K; R_f = 0.55 (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 benzene at room temperature.

S3. Refinement

All H atoms were positioned geometrically and refined using a riding model, with C–H = 0.93 Å for aromatic H atoms and with U_{iso}(H) = 1.2U_{eq}(C) for aromatic H atoms.

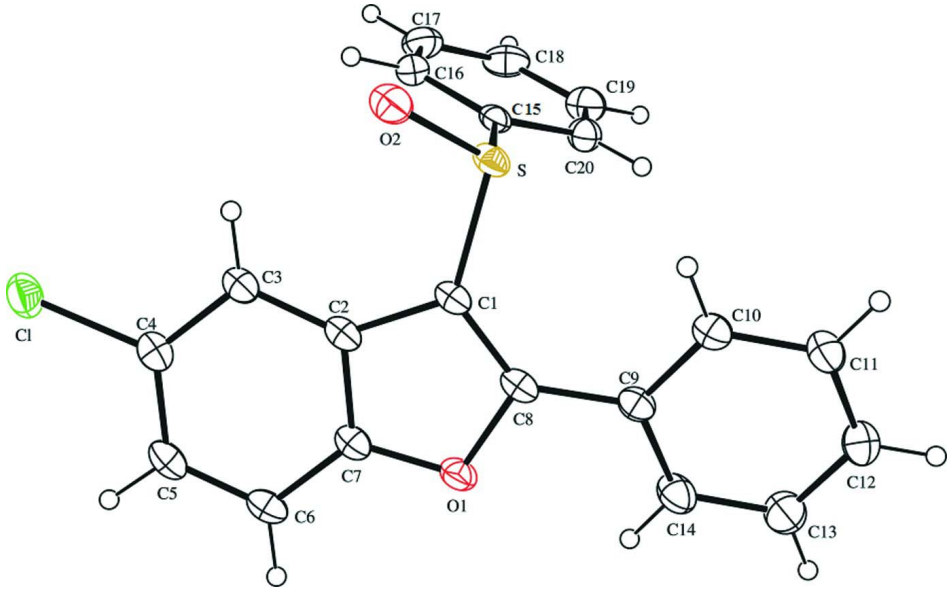

Figure 1

The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are presented as a small spheres of arbitrary radius.

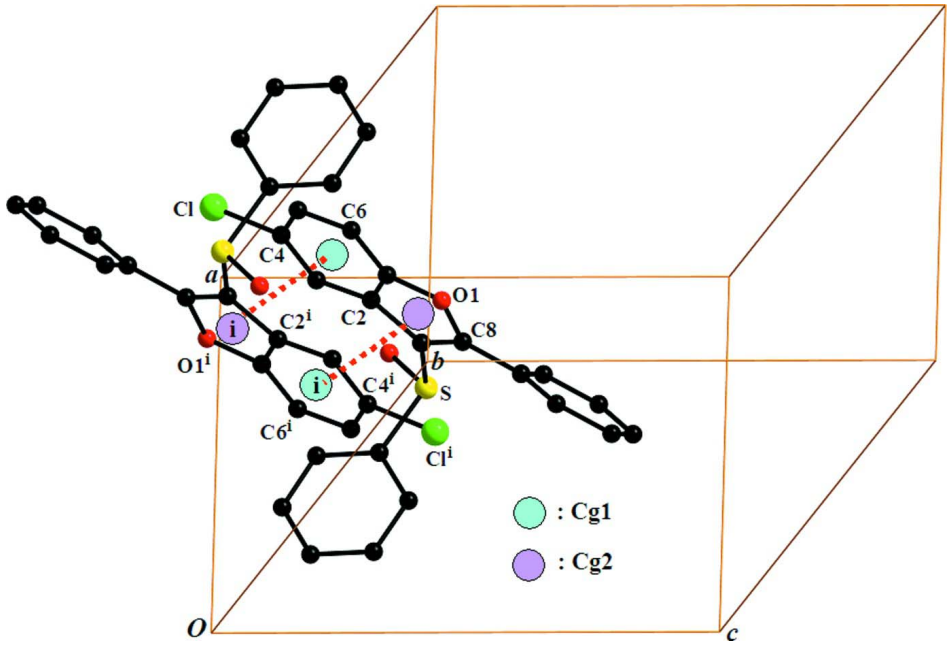

Figure 2

The π–π interactions (dotted lines) in the title compound. Cg denotes the ring centroids. [Symmetry code: (i) - x + 1, - y + 1, -z.]

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Crystal data

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$M_r = 352.81$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.2726$ (5) Å
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$c = 11.3811$ (6) Å
$\alpha = 73.360$ (1)°
$\beta = 81.630$ (1)°
$\gamma = 70.757$ (1)°
$V = 800.25$ (8) Å^3
$Z = 2$
$F(000) = 364$
$D_x = 1.464$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4091 reflections
$\theta = 2.4$–27.4°
$\mu = 0.38$ mm^{-1}
$T = 273$ K
Block, colorless
0.24 × 0.15 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.0 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1999)
$T_{min} = 0.915$, $T_{max} = 0.963$
6930 measured reflections
3428 independent reflections
2843 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 27.0°$, $\theta_{min} = 1.9°$
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.093$
$S = 1.06$
3428 reflections
217 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
$w = 1/[\sigma^2(F_o^2) + (0.0423P)^2 + 0.2959P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S	0.60764 (5)	0.10727 (5)	0.36530 (4)	0.03141 (12)
Cl	1.01634 (7)	0.23915 (6)	−0.11687 (4)	0.04741 (15)
O1	0.50587 (16)	0.56115 (13)	0.20476 (11)	0.0353 (3)

O2	0.78018 (15)	0.00894 (15)	0.33104 (13)	0.0442 (3)
C1	0.5863 (2)	0.29999 (19)	0.27413 (15)	0.0299 (4)
C2	0.6787 (2)	0.33818 (19)	0.15726 (15)	0.0308 (4)
C3	0.7989 (2)	0.2542 (2)	0.08331 (16)	0.0336 (4)
H3	0.8357	0.1460	0.1047	0.040*
C4	0.8610 (2)	0.3395 (2)	−0.02336 (17)	0.0368 (4)
C5	0.8064 (3)	0.5023 (2)	−0.05899 (17)	0.0415 (4)
H5	0.8517	0.5545	−0.1317	0.050*
C6	0.6860 (3)	0.5857 (2)	0.01297 (17)	0.0401 (4)
H6	0.6476	0.6939	−0.0094	0.048*
C7	0.6246 (2)	0.5006 (2)	0.12049 (16)	0.0331 (4)
C8	0.4853 (2)	0.4365 (2)	0.29930 (15)	0.0314 (4)
C9	0.3623 (2)	0.4786 (2)	0.39888 (16)	0.0325 (4)
C10	0.3580 (3)	0.3731 (2)	0.51308 (17)	0.0391 (4)
H10	0.4347	0.2730	0.5270	0.047*
C11	0.2402 (3)	0.4167 (2)	0.60561 (18)	0.0435 (5)
H11	0.2382	0.3455	0.6814	0.052*
C12	0.1258 (3)	0.5644 (3)	0.58682 (19)	0.0465 (5)
H12	0.0467	0.5927	0.6494	0.056*
C13	0.1293 (3)	0.6706 (3)	0.4743 (2)	0.0515 (5)
H13	0.0527	0.7707	0.4612	0.062*
C14	0.2462 (2)	0.6280 (2)	0.38153 (19)	0.0445 (5)
H14	0.2477	0.7001	0.3062	0.053*
C15	0.4533 (2)	0.07047 (18)	0.29161 (15)	0.0286 (3)
C16	0.5052 (2)	−0.0090 (2)	0.20082 (17)	0.0384 (4)
H16	0.6205	−0.0422	0.1756	0.046*
C17	0.3826 (3)	−0.0381 (2)	0.14814 (19)	0.0467 (5)
H17	0.4156	−0.0906	0.0865	0.056*
C18	0.2127 (3)	0.0101 (2)	0.1865 (2)	0.0482 (5)
H18	0.1313	−0.0090	0.1498	0.058*
C19	0.1615 (2)	0.0868 (2)	0.2791 (2)	0.0444 (5)
H19	0.0464	0.1177	0.3053	0.053*
C20	0.2819 (2)	0.1173 (2)	0.33250 (17)	0.0352 (4)
H20	0.2488	0.1684	0.3950	0.042*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0263 (2)	0.0282 (2)	0.0325 (2)	−0.00846 (16)	−0.00388 (16)	0.00459 (16)
Cl	0.0486 (3)	0.0557 (3)	0.0388 (3)	−0.0223 (2)	0.0101 (2)	−0.0119 (2)
O1	0.0398 (7)	0.0282 (6)	0.0336 (7)	−0.0129 (5)	−0.0012 (5)	0.0010 (5)
O2	0.0248 (6)	0.0357 (7)	0.0569 (9)	−0.0031 (5)	−0.0028 (6)	0.0047 (6)
C1	0.0275 (8)	0.0290 (8)	0.0309 (9)	−0.0122 (7)	−0.0048 (7)	0.0017 (7)
C2	0.0285 (8)	0.0323 (9)	0.0303 (8)	−0.0141 (7)	−0.0056 (7)	0.0017 (7)
C3	0.0313 (9)	0.0341 (9)	0.0338 (9)	−0.0136 (7)	−0.0027 (7)	−0.0014 (7)
C4	0.0339 (9)	0.0427 (10)	0.0338 (9)	−0.0160 (8)	−0.0011 (7)	−0.0051 (8)
C5	0.0477 (11)	0.0449 (11)	0.0312 (9)	−0.0245 (9)	0.0013 (8)	0.0017 (8)
C6	0.0480 (11)	0.0323 (9)	0.0368 (10)	−0.0185 (8)	−0.0035 (8)	0.0041 (8)

C7	0.0335 (9)	0.0327 (9)	0.0311 (9)	−0.0136 (7)	−0.0034 (7)	−0.0001 (7)
C8	0.0316 (9)	0.0312 (9)	0.0303 (9)	−0.0150 (7)	−0.0063 (7)	0.0026 (7)
C9	0.0312 (9)	0.0331 (9)	0.0339 (9)	−0.0138 (7)	−0.0043 (7)	−0.0039 (7)
C10	0.0450 (10)	0.0333 (9)	0.0360 (10)	−0.0122 (8)	−0.0019 (8)	−0.0039 (8)
C11	0.0494 (11)	0.0456 (11)	0.0334 (10)	−0.0181 (9)	0.0022 (8)	−0.0047 (8)
C12	0.0405 (11)	0.0521 (12)	0.0458 (11)	−0.0154 (9)	0.0082 (9)	−0.0147 (9)
C13	0.0420 (11)	0.0415 (11)	0.0579 (13)	−0.0026 (9)	0.0036 (10)	−0.0075 (10)
C14	0.0393 (10)	0.0386 (10)	0.0432 (11)	−0.0077 (8)	0.0002 (8)	0.0024 (8)
C15	0.0277 (8)	0.0218 (8)	0.0300 (8)	−0.0077 (6)	−0.0005 (6)	0.0025 (6)
C16	0.0398 (10)	0.0278 (9)	0.0408 (10)	−0.0072 (7)	0.0059 (8)	−0.0060 (7)
C17	0.0639 (14)	0.0327 (10)	0.0452 (11)	−0.0148 (9)	−0.0050 (10)	−0.0115 (8)
C18	0.0533 (12)	0.0337 (10)	0.0619 (13)	−0.0165 (9)	−0.0204 (10)	−0.0063 (9)
C19	0.0290 (9)	0.0413 (11)	0.0610 (13)	−0.0122 (8)	−0.0040 (9)	−0.0075 (9)
C20	0.0297 (9)	0.0335 (9)	0.0398 (10)	−0.0094 (7)	0.0006 (7)	−0.0069 (8)

Geometric parameters (Å, º)

S—O2	1.4898 (13)	C10—C11	1.383 (3)
S—C1	1.7782 (16)	C10—H10	0.9300
S—C15	1.7953 (17)	C11—C12	1.376 (3)
Cl—C4	1.7471 (19)	C11—H11	0.9300
O1—C7	1.371 (2)	C12—C13	1.384 (3)
O1—C8	1.3850 (19)	C12—H12	0.9300
C1—C8	1.365 (2)	C13—C14	1.378 (3)
C1—C2	1.447 (2)	C13—H13	0.9300
C2—C3	1.393 (2)	C14—H14	0.9300
C2—C7	1.396 (2)	C15—C16	1.383 (3)
C3—C4	1.384 (2)	C15—C20	1.391 (2)
C3—H3	0.9300	C16—C17	1.385 (3)
C4—C5	1.400 (3)	C16—H16	0.9300
C5—C6	1.376 (3)	C17—C18	1.374 (3)
C5—H5	0.9300	C17—H17	0.9300
C6—C7	1.386 (2)	C18—C19	1.384 (3)
C6—H6	0.9300	C18—H18	0.9300
C8—C9	1.460 (2)	C19—C20	1.382 (3)
C9—C14	1.395 (3)	C19—H19	0.9300
C9—C10	1.395 (2)	C20—H20	0.9300
O2—S—C1	106.48 (8)	C11—C10—H10	119.8
O2—S—C15	107.16 (8)	C9—C10—H10	119.8
C1—S—C15	97.09 (7)	C12—C11—C10	120.71 (18)
C7—O1—C8	106.97 (13)	C12—C11—H11	119.6
C8—C1—C2	107.66 (14)	C10—C11—H11	119.6
C8—C1—S	127.92 (13)	C11—C12—C13	119.62 (19)
C2—C1—S	124.42 (13)	C11—C12—H12	120.2
C3—C2—C7	119.65 (15)	C13—C12—H12	120.2
C3—C2—C1	135.69 (16)	C14—C13—C12	120.05 (19)
C7—C2—C1	104.66 (15)	C14—C13—H13	120.0

C4—C3—C2	116.86 (16)	C12—C13—H13	120.0
C4—C3—H3	121.6	C13—C14—C9	121.03 (18)
C2—C3—H3	121.6	C13—C14—H14	119.5
C3—C4—C5	122.95 (18)	C9—C14—H14	119.5
C3—C4—Cl	118.45 (15)	C16—C15—C20	121.14 (17)
C5—C4—Cl	118.60 (14)	C16—C15—S	120.62 (13)
C6—C5—C4	120.37 (16)	C20—C15—S	118.17 (13)
C6—C5—H5	119.8	C15—C16—C17	118.89 (18)
C4—C5—H5	119.8	C15—C16—H16	120.6
C5—C6—C7	116.83 (17)	C17—C16—H16	120.6
C5—C6—H6	121.6	C18—C17—C16	120.34 (19)
C7—C6—H6	121.6	C18—C17—H17	119.8
O1—C7—C6	125.88 (16)	C16—C17—H17	119.8
O1—C7—C2	110.78 (14)	C17—C18—C19	120.68 (19)
C6—C7—C2	123.33 (17)	C17—C18—H18	119.7
C1—C8—O1	109.91 (15)	C19—C18—H18	119.7
C1—C8—C9	135.11 (15)	C20—C19—C18	119.80 (19)
O1—C8—C9	114.96 (15)	C20—C19—H19	120.1
C14—C9—C10	118.28 (17)	C18—C19—H19	120.1
C14—C9—C8	119.98 (16)	C19—C20—C15	119.13 (18)
C10—C9—C8	121.73 (16)	C19—C20—H20	120.4
C11—C10—C9	120.32 (18)	C15—C20—H20	120.4

O2—S—C1—C8	−154.75 (16)	C7—O1—C8—C1	−1.18 (18)
C15—S—C1—C8	94.95 (17)	C7—O1—C8—C9	180.00 (14)
O2—S—C1—C2	24.56 (16)	C1—C8—C9—C14	−162.7 (2)
C15—S—C1—C2	−85.74 (15)	O1—C8—C9—C14	15.7 (2)
C8—C1—C2—C3	179.49 (19)	C1—C8—C9—C10	18.0 (3)
S—C1—C2—C3	0.1 (3)	O1—C8—C9—C10	−163.57 (16)
C8—C1—C2—C7	0.36 (18)	C14—C9—C10—C11	0.5 (3)
S—C1—C2—C7	−179.07 (12)	C8—C9—C10—C11	179.81 (17)
C7—C2—C3—C4	1.3 (2)	C9—C10—C11—C12	−0.1 (3)
C1—C2—C3—C4	−177.78 (18)	C10—C11—C12—C13	−0.3 (3)
C2—C3—C4—C5	−1.0 (3)	C11—C12—C13—C14	0.3 (3)
C2—C3—C4—Cl	178.31 (13)	C12—C13—C14—C9	0.0 (3)
C3—C4—C5—C6	0.2 (3)	C10—C9—C14—C13	−0.5 (3)
Cl—C4—C5—C6	−179.13 (15)	C8—C9—C14—C13	−179.77 (19)
C4—C5—C6—C7	0.4 (3)	O2—S—C15—C16	−12.69 (15)
C8—O1—C7—C6	−178.23 (17)	C1—S—C15—C16	97.05 (14)
C8—O1—C7—C2	1.43 (18)	O2—S—C15—C20	164.24 (13)
C5—C6—C7—O1	179.52 (17)	C1—S—C15—C20	−86.02 (14)
C5—C6—C7—C2	−0.1 (3)	C20—C15—C16—C17	1.7 (3)
C3—C2—C7—O1	179.59 (14)	S—C15—C16—C17	178.56 (13)
C1—C2—C7—O1	−1.11 (19)	C15—C16—C17—C18	−0.6 (3)
C3—C2—C7—C6	−0.7 (3)	C16—C17—C18—C19	−0.7 (3)
C1—C2—C7—C6	178.56 (17)	C17—C18—C19—C20	0.9 (3)
C2—C1—C8—O1	0.50 (19)	C18—C19—C20—C15	0.2 (3)
S—C1—C8—O1	179.91 (12)	C16—C15—C20—C19	−1.6 (2)

C2—C1—C8—C9	178.98 (18)	S—C15—C20—C19	−178.46 (13)
S—C1—C8—C9	−1.6 (3)		