Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-Iodo-7-methyl-3-methylsulfinyl-2-phenyl-1-benzofuran

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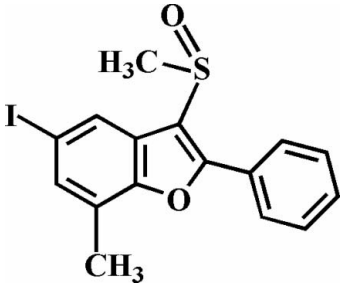
Received 14 May 2008; accepted 15 May 2008

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.020; wR factor = 0.054; data-to-parameter ratio = 15.7.

The title compound, $C_{16}H_{13}IO_2S$, was prepared by the oxidation of 5-iodo-7-methyl-3-methylsulfanyl-2-phenyl-1-benzofuran with 3-chloroperoxybenzoic acid. The phenyl ring makes a dihedral angle of 27.17 (9)° with the plane of the benzofuran fragment, with the O atom and the methyl group of the methylsulfinyl substituent lying on opposite sides of this plane. The crystal structure exhibits intermolecular C—H···I interactions, and an I···O halogen bond of 3.107 (2) Å with a nearly linear C—I···O angle of 173.73 (6)°.

Related literature

For the crystal structures of similar 5-halo-3-methylsulfinyl-2-phenyl-1-benzofuran compounds, see: Choi *et al.* (2007*a*,*b*). For a review of halogen bonding, see: Politzer *et al.* (2007).



Experimental

Crystal data

$C_{16}H_{13}IO_2S$
M_r = 396.22
Monoclinic, $P2_1/c$
a = 10.385 (5) Å
b = 17.174 (8) Å
c = 8.943 (4) Å
β = 112.847 (7)°
V = 1469.9 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.32 mm^{-1}
T = 173 (2) K
0.40 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
T_{min} = 0.572, T_{max} = 0.623
11429 measured reflections
2877 independent reflections
2706 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.053
S = 1.06
2877 reflections
183 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.66 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···I^{i}	0.95	3.06	3.954 (3)	157

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

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: ZL2118).

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supporting information

Acta Cryst. (2008). E**64**, o1116 [doi:10.1107/S1600536808014694]

5-Iodo-7-methyl-3-methylsulfinyl-2-phenyl-1-benzofuran

Hong Dae Choi, Pil Ja Seo, Byung Ki Kim, Byeng Wha Son and Uk Lee

S1. Comment

This work is related to our previous communications on the synthesis and structure of 5-halo-3-methylsulfinyl-2-phenyl-1-benzofuran analogues, viz. 5-bromo-3-methylsulfinyl-2-phenyl-1-benzofuran (Choi *et al.*, 2007*a*) and 5-iodo-3-methylsulfinyl-2-phenyl-1-benzofuran (Choi *et al.*, 2007*b*). Here we report the crystal structure of the title compound, 5-iodo-7-methyl-3-methylsulfinyl-2-phenyl-1-benzofuran (Fig. 1).

The benzofuran unit is essentially planar, with a mean deviation of 0.013 Å from the least-squares plane defined by the nine constituent atoms. The phenyl ring (C9-C14) makes a dihedral angle of 27.17 (9)° with the plane of the benzofuran fragment. The molecular packing (Fig. 2) is stabilized by intermolecular C—H···I interactions (Table 1), and by an I···O halogen bond (Politzer *et al.*, 2007) between the iodine atom and the oxygen of a neighbouring S═O unit, with an I···O2^{i} distance of 3.107 (2) Å (symmetry code as in Fig. 2).

S2. Experimental

77% 3-chloroperoxybenzoic acid (247 mg, 1.1 mmol) was added in small portions to a stirred solution of 5-iodo-7-methyl-3-methylsulfanyl-2-phenyl-1-benzofuran (380 mg, 1.0 mmol) in dichloromethane (30 ml) at 273 K. After being stirred for 4 h at room temperature, 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, 1:1 v/v) to afford the title compound as a colorless solid [yield 79%, m.p. 433-434 K; R_f = 0.51 (hexane-ethyl acetate, 1:1 v/v)]. Single crystals suitable for X-ray diffraction were prepared by evaporation of a solution of the title compound in tetrahydrofuran at room temperature. Spectroscopic analysis: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.53 (s, 3H), 3.11 (s, 3H), 7.49-7.58 (m, 4H), 7.83 (dd, J = 8.04 Hz and 1.84 Hz, 2H), 8.39 (s, 1H); EI-MS 396 [M^+].

S3. Refinement

All H atoms were geometrically positioned and refined using a riding model, with C-H = 0.95 Å for aromatic H atoms, 0.98 Å for methyl H atoms, respectively, and with Uiso(H) = 1.2Ueq(C) for aromatic H atoms and 1.5Ueq(C) for methyl H atoms. The highest peak in the difference map is 0.98 Å from I and the largest hole is 0.92 Å from I.

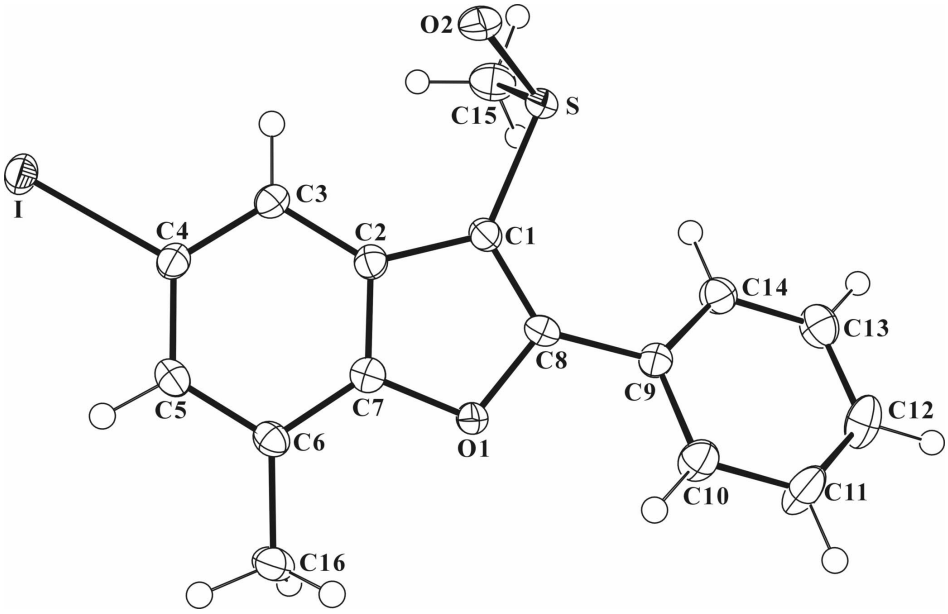

Figure 1

The molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.

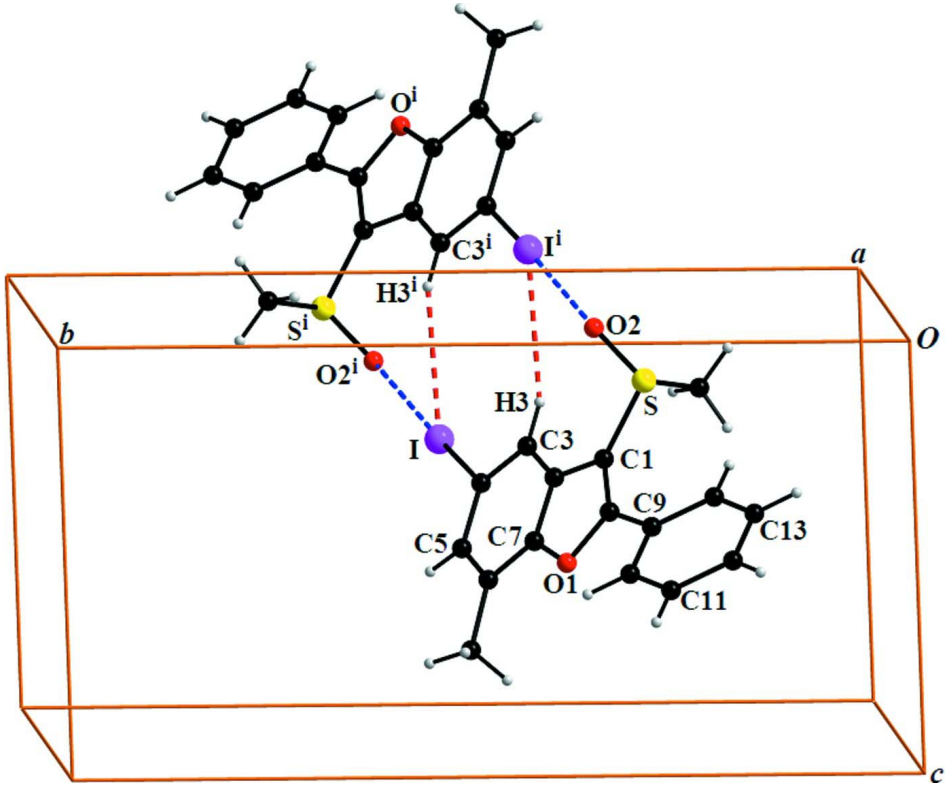

Figure 2

C—H···I interaction and I···O halogen bond (dotted lines) in the title compound. [Symmetry code: (i) -x, -y+1, -z.]

5-Iodo-7-methyl-3-methylsulfinyl-2-phenyl-1-benzofuran

Crystal data

$C_{16}H_{13}IO_2S$
$M_r = 396.22$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 10.385\ (5)$ Å
$b = 17.174\ (8)$ Å
$c = 8.943\ (4)$ Å
$\beta = 112.847\ (7)°$
$V = 1469.9\ (12)$ Å^3
$Z = 4$
$F(000) = 776$
$D_x = 1.790$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9956 reflections
$\theta = 2.1–28.4°$
$\mu = 2.32$ mm^{-1}
$T = 173$ K
Block, colorless
0.40 × 0.20 × 0.20 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, 2000)
$T_{min} = 0.572$, $T_{max} = 0.623$
11429 measured reflections
2877 independent reflections
2706 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.4°$
$h = -12\rightarrow12$
$k = -21\rightarrow21$
$l = -11\rightarrow11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.020$
$wR(F^2) = 0.053$
$S = 1.07$
2877 reflections
183 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0278P)^2 + 0.8832P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.66$ 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}
I	−0.043288 (13)	0.558358 (8)	0.210981 (16)	0.02333 (7)
S	0.36162 (5)	0.29269 (3)	0.14788 (6)	0.02051 (11)
O1	0.50724 (14)	0.38231 (8)	0.59241 (16)	0.0184 (3)
O2	0.30733 (17)	0.35243 (9)	0.01551 (18)	0.0280 (3)

C1	0.3955 (2)	0.34115 (11)	0.3340 (2)	0.0186 (4)
C2	0.3154 (2)	0.40433 (12)	0.3616 (2)	0.0185 (4)
C3	0.1901 (2)	0.44237 (11)	0.2685 (3)	0.0199 (4)
H3	0.1385	0.4291	0.1581	0.024*
C4	0.1451 (2)	0.50027 (12)	0.3454 (2)	0.0203 (4)
C5	0.2211 (2)	0.52118 (12)	0.5084 (2)	0.0211 (4)
H5	0.1866	0.5614	0.5556	0.025*
C6	0.3460 (2)	0.48423 (11)	0.6021 (2)	0.0195 (4)
C7	0.3883 (2)	0.42659 (12)	0.5218 (2)	0.0182 (4)
C8	0.5095 (2)	0.32999 (11)	0.4756 (2)	0.0180 (4)
C9	0.6257 (2)	0.27448 (12)	0.5307 (2)	0.0184 (4)
C10	0.7494 (2)	0.29442 (13)	0.6599 (3)	0.0267 (5)
H10	0.7591	0.3445	0.7082	0.032*
C11	0.8579 (2)	0.24128 (14)	0.7177 (3)	0.0357 (6)
H11	0.9417	0.2550	0.8061	0.043*
C12	0.8452 (2)	0.16800 (14)	0.6474 (3)	0.0333 (5)
H12	0.9199	0.1317	0.6875	0.040*
C13	0.7229 (2)	0.14820 (13)	0.5185 (3)	0.0284 (5)
H13	0.7144	0.0984	0.4693	0.034*
C14	0.6129 (2)	0.20047 (12)	0.4608 (3)	0.0242 (4)
H14	0.5287	0.1861	0.3738	0.029*
C15	0.4267 (2)	0.50275 (13)	0.7788 (3)	0.0265 (5)
H15A	0.4092	0.4624	0.8460	0.040*
H15B	0.3967	0.5534	0.8043	0.040*
H15C	0.5268	0.5046	0.8010	0.040*
C16	0.2127 (2)	0.23678 (14)	0.1402 (3)	0.0310 (5)
H16A	0.1375	0.2721	0.1369	0.046*
H16B	0.2391	0.2037	0.2369	0.046*
H16C	0.1803	0.2041	0.0428	0.046*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I	0.01881 (9)	0.02267 (10)	0.02713 (10)	0.00497 (5)	0.00739 (7)	0.00177 (5)
S	0.0221 (3)	0.0235 (3)	0.0161 (2)	0.0048 (2)	0.00748 (19)	−0.00083 (18)
O1	0.0190 (7)	0.0184 (7)	0.0170 (6)	0.0028 (5)	0.0060 (6)	−0.0001 (5)
O2	0.0330 (9)	0.0325 (8)	0.0175 (7)	0.0065 (7)	0.0088 (6)	0.0050 (6)
C1	0.0197 (9)	0.0193 (10)	0.0179 (9)	0.0009 (8)	0.0084 (8)	−0.0004 (7)
C2	0.0194 (9)	0.0184 (9)	0.0193 (9)	0.0006 (8)	0.0094 (8)	0.0015 (7)
C3	0.0199 (10)	0.0203 (10)	0.0187 (10)	0.0017 (8)	0.0067 (8)	0.0017 (7)
C4	0.0170 (9)	0.0227 (10)	0.0215 (10)	0.0023 (8)	0.0077 (8)	0.0038 (8)
C5	0.0248 (10)	0.0186 (10)	0.0226 (10)	0.0023 (8)	0.0123 (9)	−0.0007 (8)
C6	0.0233 (10)	0.0180 (9)	0.0193 (10)	−0.0003 (8)	0.0105 (8)	0.0006 (7)
C7	0.0185 (10)	0.0169 (9)	0.0194 (10)	−0.0004 (8)	0.0075 (8)	0.0025 (7)
C8	0.0205 (10)	0.0174 (9)	0.0181 (9)	−0.0016 (8)	0.0096 (8)	−0.0008 (7)
C9	0.0177 (9)	0.0206 (10)	0.0186 (9)	0.0003 (8)	0.0090 (8)	0.0040 (7)
C10	0.0219 (11)	0.0215 (10)	0.0324 (12)	−0.0011 (9)	0.0057 (9)	−0.0006 (9)
C11	0.0186 (11)	0.0308 (12)	0.0452 (14)	0.0026 (9)	−0.0013 (10)	0.0021 (10)

C12	0.0233 (11)	0.0274 (12)	0.0478 (14)	0.0080 (9)	0.0121 (11)	0.0091 (10)
C13	0.0324 (12)	0.0209 (10)	0.0338 (12)	0.0036 (9)	0.0150 (10)	0.0013 (9)
C14	0.0251 (11)	0.0232 (10)	0.0234 (10)	0.0010 (9)	0.0086 (9)	−0.0003 (8)
C15	0.0320 (12)	0.0269 (11)	0.0190 (10)	0.0020 (9)	0.0080 (9)	−0.0031 (8)
C16	0.0315 (12)	0.0311 (12)	0.0278 (11)	−0.0070 (10)	0.0087 (10)	−0.0055 (9)

Geometric parameters (Å, º)

I—C4	2.107 (2)	C8—C9	1.465 (3)
I—O2^{i}	3.107 (2)	C9—C10	1.396 (3)
S—O2	1.501 (2)	C9—C14	1.400 (3)
S—C1	1.770 (2)	C10—C11	1.385 (3)
S—C16	1.799 (2)	C10—H10	0.9500
O1—C7	1.377 (2)	C11—C12	1.390 (4)
O1—C8	1.385 (2)	C11—H11	0.9500
C1—C8	1.370 (3)	C12—C13	1.386 (3)
C1—C2	1.446 (3)	C12—H12	0.9500
C2—C7	1.389 (3)	C13—C14	1.385 (3)
C2—C3	1.404 (3)	C13—H13	0.9500
C3—C4	1.389 (3)	C14—H14	0.9500
C3—H3	0.9500	C15—H15A	0.9800
C4—C5	1.409 (3)	C15—H15B	0.9800
C5—C6	1.394 (3)	C15—H15C	0.9800
C5—H5	0.9500	C16—H16A	0.9800
C6—C7	1.391 (3)	C16—H16B	0.9800
C6—C15	1.507 (3)	C16—H16C	0.9800
C4—I—O2^{i}	173.73 (6)	C10—C9—C8	119.45 (19)
O2—S—C1	107.27 (10)	C14—C9—C8	121.16 (18)
O2—S—C16	106.30 (11)	C11—C10—C9	120.1 (2)
C1—S—C16	98.10 (10)	C11—C10—H10	120.0
C7—O1—C8	106.69 (15)	C9—C10—H10	120.0
C8—C1—C2	107.15 (17)	C10—C11—C12	120.5 (2)
C8—C1—S	126.05 (16)	C10—C11—H11	119.8
C2—C1—S	126.59 (15)	C12—C11—H11	119.8
C7—C2—C3	119.48 (19)	C13—C12—C11	119.6 (2)
C7—C2—C1	105.27 (17)	C13—C12—H12	120.2
C3—C2—C1	135.23 (19)	C11—C12—H12	120.2
C4—C3—C2	116.85 (19)	C14—C13—C12	120.5 (2)
C4—C3—H3	121.6	C14—C13—H13	119.7
C2—C3—H3	121.6	C12—C13—H13	119.7
C3—C4—C5	122.24 (19)	C13—C14—C9	120.0 (2)
C3—C4—I	118.20 (15)	C13—C14—H14	120.0
C5—C4—I	119.56 (15)	C9—C14—H14	120.0
C6—C5—C4	121.56 (19)	C6—C15—H15A	109.5
C6—C5—H5	119.2	C6—C15—H15B	109.5
C4—C5—H5	119.2	H15A—C15—H15B	109.5
C5—C6—C7	114.84 (18)	C6—C15—H15C	109.5

C5—C6—C15	122.91 (19)	H15A—C15—H15C	109.5
C7—C6—C15	122.21 (19)	H15B—C15—H15C	109.5
O1—C7—C2	110.71 (17)	S—C16—H16A	109.5
O1—C7—C6	124.27 (18)	S—C16—H16B	109.5
C2—C7—C6	125.00 (19)	H16A—C16—H16B	109.5
C1—C8—O1	110.17 (17)	S—C16—H16C	109.5
C1—C8—C9	134.83 (18)	H16A—C16—H16C	109.5
O1—C8—C9	114.93 (16)	H16B—C16—H16C	109.5
C10—C9—C14	119.33 (19)		

O2—S—C1—C8	−138.12 (18)	C5—C6—C7—O1	−179.08 (18)
C16—S—C1—C8	111.9 (2)	C15—C6—C7—O1	−1.3 (3)
O2—S—C1—C2	36.1 (2)	C5—C6—C7—C2	−0.8 (3)
C16—S—C1—C2	−73.9 (2)	C15—C6—C7—C2	176.9 (2)
C8—C1—C2—C7	−0.4 (2)	C2—C1—C8—O1	0.0 (2)
S—C1—C2—C7	−175.49 (15)	S—C1—C8—O1	175.08 (14)
C8—C1—C2—C3	−179.1 (2)	C2—C1—C8—C9	176.7 (2)
S—C1—C2—C3	5.9 (3)	S—C1—C8—C9	−8.2 (3)
C7—C2—C3—C4	−1.1 (3)	C7—O1—C8—C1	0.5 (2)
C1—C2—C3—C4	177.4 (2)	C7—O1—C8—C9	−176.98 (16)
C2—C3—C4—C5	0.8 (3)	C1—C8—C9—C10	157.6 (2)
C2—C3—C4—I	−179.53 (14)	O1—C8—C9—C10	−25.8 (3)
C3—C4—C5—C6	−0.5 (3)	C1—C8—C9—C14	−25.2 (3)
I—C4—C5—C6	179.82 (15)	O1—C8—C9—C14	151.38 (18)
C4—C5—C6—C7	0.5 (3)	C14—C9—C10—C11	0.0 (3)
C4—C5—C6—C15	−177.3 (2)	C8—C9—C10—C11	177.3 (2)
C8—O1—C7—C2	−0.7 (2)	C9—C10—C11—C12	0.4 (4)
C8—O1—C7—C6	177.71 (19)	C10—C11—C12—C13	0.0 (4)
C3—C2—C7—O1	179.63 (17)	C11—C12—C13—C14	−0.8 (4)
C1—C2—C7—O1	0.7 (2)	C12—C13—C14—C9	1.2 (3)
C3—C2—C7—C6	1.2 (3)	C10—C9—C14—C13	−0.8 (3)
C1—C2—C7—C6	−177.73 (19)	C8—C9—C14—C13	−178.01 (19)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···I^{i}	0.95	3.06	3.954 (3)	157

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