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5-Iodo-2-methyl-3-(4-methylphenylsulfanyl)-1-benzofuran

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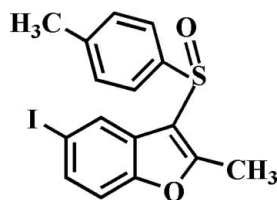
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Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.027; wR factor = 0.068; data-to-parameter ratio = 19.1.

In the title compound, $\text{C}_{16}\text{H}_{13}\text{IO}_2\text{S}$, the 4-methylphenyl ring makes a dihedral angle of $88.36(7)^\circ$ with the mean plane [r.m.s. deviation = $0.006(2)$ Å] of the benzofuran fragment. In the crystal, molecules are linked by weak $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds and an $\text{I}\cdots\text{O}$ contact [$3.103(2)$ Å]. The crystal structure also exhibits slipped $\pi-\pi$ interactions between the benzene and furan rings of neighbouring molecules [centroid-centroid distance = $3.659(2)$ Å].

Related literature

For background information and the crystal structures of related compounds, see: Choi *et al.* (2007, 2010*a,b*). For a review of halogen bonding, see: Politzer *et al.* (2007).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{13}\text{IO}_2\text{S}$
 $M_r = 396.22$
 Monoclinic, $P2_1/c$
 $a = 14.3793(3)$ Å
 $b = 11.4519(2)$ Å
 $c = 9.6419(2)$ Å
 $\beta = 107.854(1)^\circ$
 $V = 1511.27(5)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 2.26$ mm⁻¹
 $T = 173$ K
 $0.22 \times 0.20 \times 0.08$ mm

Data collection

Bruker SMART APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2009)
 $T_{\min} = 0.638$, $T_{\max} = 0.846$
 13752 measured reflections
 3490 independent reflections
 2900 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.027$
 $wR(F^2) = 0.068$
 $S = 1.05$
 3490 reflections
 183 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.62$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.18$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C5}-\text{H5}\cdots\text{O1}^i$	0.95	2.52	3.459(3)	170
$\text{C9}-\text{H9A}\cdots\text{O2}^{ii}$	0.98	2.52	3.325(3)	139
$\text{C15}-\text{H15}\cdots\text{O2}^{ii}$	0.95	2.58	3.499(3)	162

Symmetry codes: (i) $-x + 2, y + \frac{1}{2}, -z + \frac{3}{2}$; (ii) $x, -y + \frac{1}{2}, z + \frac{1}{2}$.

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: ZL2489).

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supporting information

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5-Iodo-2-methyl-3-(4-methylphenylsulfinyl)-1-benzofuran**Hong Dae Choi, Pil Ja Seo and Uk Lee****S1. Comment**

As a part of our ongoing study of 5-iodo-2-methyl-1-benzofuran derivatives containing 3-phenylsulfinyl (Choi *et al.*, 2007), 3-(4-fluorophenylsulfinyl) (Choi *et al.*, 2010a), and 3-(4-fluorophenylsulfonyl) (Choi *et al.*, 2010b) substituents, 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 0.006 (2) Å from the least-squares plane defined by the nine constituent atoms. The dihedral angle between the 4-methylphenyl ring and the mean plane of the benzofuran ring is 88.36 (7)°. In the crystal structure (Fig. 2), molecules are connected by weak C–H···O hydrogen bonds (Table 1), and by an I···O halogen-bond between the iodine and the oxygen of the S=O unit [I···Oⁱ = 3.103 (2) Å, C4–I···Oⁱ = 166.89 (8)°] (Politzer *et al.*, 2007). The crystal packing (Fig. 3) also exhibits slipped π – π interactions between the benzene and furan rings of neighbouring molecules, with a Cg1···Cg2^{vi} distance of 3.659 (2) Å and an interplanar distance of 3.321 (2) Å resulting in a slippage of 1.536 (2) Å (Cg1 and Cg2 are the centroids of the C2–C7 benzene ring and the C1/C2/C7/O1/C8 furan ring, respectively).

S2. Experimental

3-Chloroperoxybenzoic acid (77%, 224 mg, 1.0 mmol) was added in small portions to a stirred solution of 5-iodo-2-methyl-3-(4-methylphenylsulfanyl)-1-benzofuran (342 mg, 0.9 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, 2:1 v/v) to afford the title compound as a colorless solid [yield 78%, m.p. 456–457 K; R_f = 0.48 (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 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 and 0.98 Å for methyl H atoms. $U_{iso}(H) = 1.2U_{eq}(C)$ for aryl and $1.5U_{eq}(C)$ for methyl H atoms. The positions of methyl hydrogens were optimized rotationally.

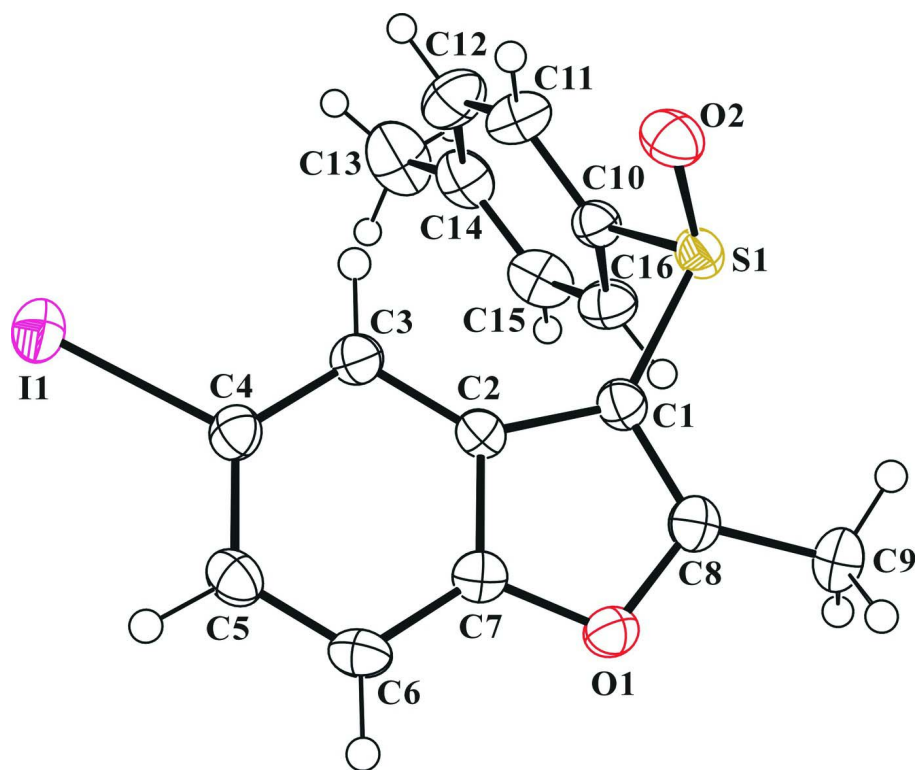


Figure 1

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.

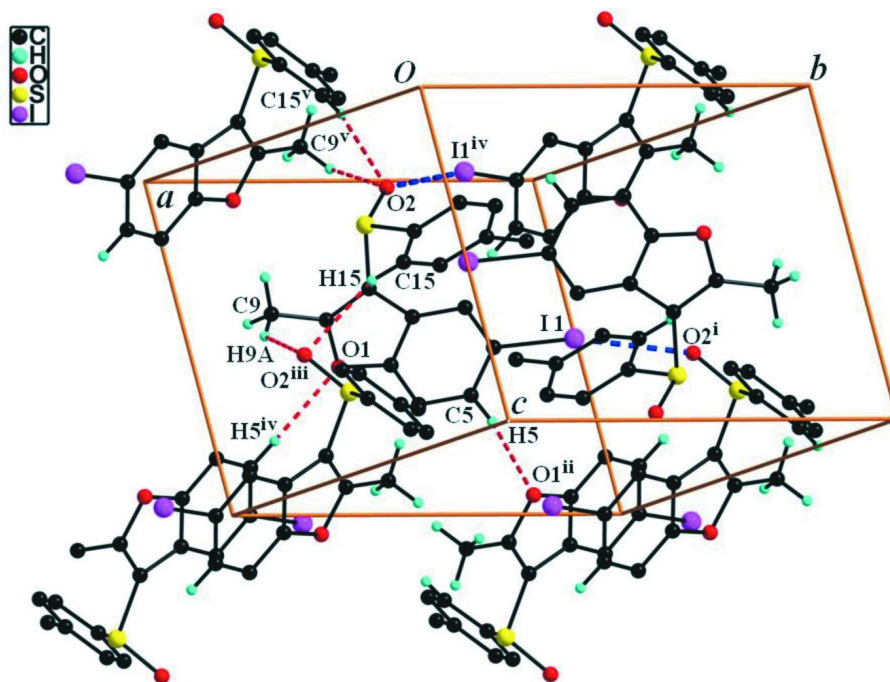


Figure 2

A view of the C–H···O and I–O interactions (dotted lines) in the crystal structure of the title compound. H atoms non-participating in hydrogen-bonding were omitted for clarity. [Symmetry codes: (i) $x, -y + 3/2, z + 1/2$ (ii) $-x + 2, y + 1/2, -z + 3/2$ (iii) $x, -y + 1/2, z + 1/2$ (iv) $x, -y + 3/2, z - 1/2$ (v) $x, -y + 1/2, z - 1/2$.]

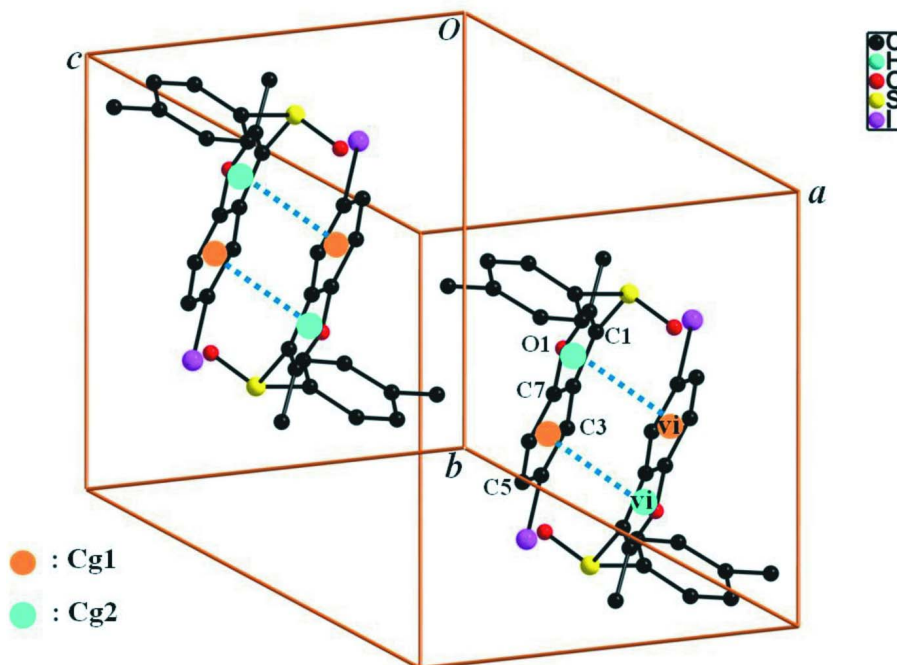


Figure 3

A view of the π – π interactions (dotted lines) in the crystal structure of the title compound. All H atoms were omitted for clarity. [Symmetry codes: (vi) $-x + 2, -y + 1, -z + 1$.]

5-Iodo-2-methyl-3-(4-methylphenylsulfinyl)-1-benzofuran

Crystal data

C₁₆H₁₃IO₂S $M_r = 396.22$ Monoclinic, $P2_1/c$ $a = 14.3793$ (3) Å $b = 11.4519$ (2) Å $c = 9.6419$ (2) Å $\beta = 107.854$ (1)° $V = 1511.27$ (5) Å³ $Z = 4$ $F(000) = 776$ $D_x = 1.741$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 5240 reflections

 $\theta = 2.9$ – 27.5 ° $\mu = 2.26$ mm⁻¹ $T = 173$ K

Block, colourless

 $0.22 \times 0.20 \times 0.08$ 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 scansAbsorption correction: multi-scan
(*SADABS*; Bruker, 2009) $T_{\min} = 0.638$, $T_{\max} = 0.846$

13752 measured reflections

3490 independent reflections

2900 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.033$ $\theta_{\max} = 27.5$ °, $\theta_{\min} = 1.5$ ° $h = -18$ → 18 $k = -14$ → 12 $l = -12$ → 12

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.027$ $wR(F^2) = 0.068$ $S = 1.05$

3490 reflections

183 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
map

Hydrogen site location: difference Fourier map

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0302P)^2 + 0.8945P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.62$ e Å⁻³ $\Delta\rho_{\min} = -1.18$ 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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
I1	0.790469 (12)	0.834779 (16)	0.53396 (2)	0.03646 (8)
S1	0.72860 (4)	0.32747 (6)	0.20862 (7)	0.02653 (14)
O1	0.94424 (12)	0.32924 (15)	0.57306 (19)	0.0278 (4)
O2	0.73517 (13)	0.41260 (17)	0.09476 (19)	0.0351 (4)

C1	0.81709 (16)	0.3620 (2)	0.3740 (3)	0.0250 (5)
C2	0.83857 (15)	0.4706 (2)	0.4533 (2)	0.0221 (5)
C3	0.80122 (16)	0.5838 (2)	0.4364 (3)	0.0252 (5)
H3	0.7477	0.6052	0.3546	0.030*
C4	0.84537 (17)	0.6639 (2)	0.5439 (3)	0.0267 (5)
C5	0.92529 (17)	0.6352 (2)	0.6641 (3)	0.0296 (6)
H5	0.9535	0.6931	0.7351	0.036*
C6	0.96359 (16)	0.5243 (2)	0.6807 (3)	0.0287 (5)
H6	1.0185	0.5038	0.7609	0.034*
C7	0.91797 (16)	0.4444 (2)	0.5747 (3)	0.0244 (5)
C8	0.88230 (16)	0.2815 (2)	0.4494 (3)	0.0260 (5)
C9	0.89775 (19)	0.1562 (2)	0.4262 (3)	0.0334 (6)
H9A	0.8788	0.1096	0.4985	0.050*
H9B	0.9669	0.1426	0.4368	0.050*
H9C	0.8578	0.1335	0.3281	0.050*
C10	0.62215 (16)	0.3666 (2)	0.2572 (3)	0.0256 (5)
C11	0.56470 (19)	0.4576 (3)	0.1886 (3)	0.0376 (6)
H11	0.5827	0.5034	0.1188	0.045*
C12	0.47984 (19)	0.4818 (3)	0.2227 (4)	0.0446 (7)
H12	0.4397	0.5448	0.1751	0.053*
C13	0.45216 (18)	0.4171 (3)	0.3234 (3)	0.0385 (7)
C14	0.5114 (2)	0.3241 (3)	0.3901 (3)	0.0419 (7)
H14	0.4933	0.2781	0.4597	0.050*
C15	0.59568 (18)	0.2975 (3)	0.3572 (3)	0.0342 (6)
H15	0.6350	0.2330	0.4021	0.041*
C16	0.3605 (2)	0.4459 (4)	0.3621 (4)	0.0579 (10)
H16A	0.3382	0.5244	0.3269	0.087*
H16B	0.3745	0.4429	0.4681	0.087*
H16C	0.3094	0.3890	0.3162	0.087*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.04096 (11)	0.02548 (13)	0.04085 (12)	0.00036 (7)	0.00948 (8)	-0.00264 (7)
S1	0.0285 (3)	0.0261 (4)	0.0240 (3)	-0.0025 (2)	0.0065 (2)	-0.0035 (2)
O1	0.0247 (7)	0.0292 (11)	0.0284 (9)	0.0029 (7)	0.0063 (7)	0.0019 (7)
O2	0.0416 (10)	0.0375 (12)	0.0275 (9)	-0.0046 (8)	0.0127 (8)	0.0017 (8)
C1	0.0224 (10)	0.0279 (15)	0.0250 (12)	-0.0001 (9)	0.0077 (9)	-0.0026 (9)
C2	0.0185 (9)	0.0265 (14)	0.0221 (11)	-0.0019 (9)	0.0072 (8)	-0.0014 (9)
C3	0.0239 (10)	0.0255 (14)	0.0253 (12)	-0.0009 (9)	0.0063 (9)	0.0013 (9)
C4	0.0286 (11)	0.0228 (15)	0.0300 (13)	-0.0026 (9)	0.0105 (9)	0.0015 (10)
C5	0.0306 (11)	0.0314 (16)	0.0261 (13)	-0.0086 (10)	0.0076 (10)	-0.0033 (10)
C6	0.0238 (10)	0.0345 (16)	0.0247 (12)	-0.0039 (10)	0.0029 (9)	0.0026 (10)
C7	0.0215 (10)	0.0262 (14)	0.0268 (12)	-0.0002 (9)	0.0094 (9)	0.0018 (10)
C8	0.0251 (10)	0.0275 (15)	0.0283 (12)	-0.0005 (10)	0.0126 (9)	-0.0009 (10)
C9	0.0350 (12)	0.0272 (16)	0.0408 (15)	0.0028 (11)	0.0156 (11)	-0.0025 (11)
C10	0.0233 (10)	0.0251 (14)	0.0261 (12)	-0.0038 (9)	0.0041 (9)	-0.0014 (10)
C11	0.0337 (12)	0.0369 (18)	0.0401 (15)	0.0017 (11)	0.0081 (11)	0.0108 (12)

C12	0.0310 (13)	0.044 (2)	0.0555 (19)	0.0084 (12)	0.0079 (12)	0.0079 (14)
C13	0.0242 (11)	0.048 (2)	0.0411 (16)	-0.0088 (11)	0.0075 (11)	-0.0094 (13)
C14	0.0373 (14)	0.053 (2)	0.0364 (16)	-0.0125 (13)	0.0133 (12)	0.0012 (13)
C15	0.0333 (12)	0.0349 (17)	0.0324 (14)	-0.0026 (11)	0.0069 (11)	0.0055 (11)
C16	0.0314 (14)	0.079 (3)	0.065 (2)	-0.0055 (15)	0.0176 (14)	-0.0212 (19)

Geometric parameters (Å, °)

I1—C4	2.101 (2)	C8—C9	1.479 (4)
I1—O2 ⁱ	3.1034 (19)	C9—H9A	0.9800
S1—O2	1.4927 (19)	C9—H9B	0.9800
S1—C1	1.753 (2)	C9—H9C	0.9800
S1—C10	1.790 (2)	C10—C11	1.367 (4)
O1—C8	1.365 (3)	C10—C15	1.388 (4)
O1—C7	1.374 (3)	C11—C12	1.385 (4)
C1—C8	1.356 (3)	C11—H11	0.9500
C1—C2	1.443 (3)	C12—C13	1.374 (4)
C2—C3	1.394 (3)	C12—H12	0.9500
C2—C7	1.394 (3)	C13—C14	1.391 (4)
C3—C4	1.384 (3)	C13—C16	1.512 (4)
C3—H3	0.9500	C14—C15	1.379 (4)
C4—C5	1.398 (3)	C14—H14	0.9500
C5—C6	1.374 (4)	C15—H15	0.9500
C5—H5	0.9500	C16—H16A	0.9800
C6—C7	1.379 (3)	C16—H16B	0.9800
C6—H6	0.9500	C16—H16C	0.9800
C4—I1—O2 ⁱ	166.89 (8)	C8—C9—H9B	109.5
O2—S1—C1	109.25 (11)	H9A—C9—H9B	109.5
O2—S1—C10	106.33 (12)	C8—C9—H9C	109.5
C1—S1—C10	98.38 (11)	H9A—C9—H9C	109.5
C8—O1—C7	106.66 (18)	H9B—C9—H9C	109.5
C8—C1—C2	107.5 (2)	C11—C10—C15	121.1 (2)
C8—C1—S1	121.6 (2)	C11—C10—S1	120.2 (2)
C2—C1—S1	130.84 (18)	C15—C10—S1	118.5 (2)
C3—C2—C7	119.0 (2)	C10—C11—C12	118.9 (3)
C3—C2—C1	136.7 (2)	C10—C11—H11	120.5
C7—C2—C1	104.3 (2)	C12—C11—H11	120.5
C4—C3—C2	117.2 (2)	C13—C12—C11	121.8 (3)
C4—C3—H3	121.4	C13—C12—H12	119.1
C2—C3—H3	121.4	C11—C12—H12	119.1
C3—C4—C5	122.5 (2)	C12—C13—C14	118.1 (3)
C3—C4—I1	120.21 (18)	C12—C13—C16	121.3 (3)
C5—C4—I1	117.30 (19)	C14—C13—C16	120.6 (3)
C6—C5—C4	120.8 (2)	C15—C14—C13	121.3 (3)
C6—C5—H5	119.6	C15—C14—H14	119.4
C4—C5—H5	119.6	C13—C14—H14	119.4
C5—C6—C7	116.4 (2)	C14—C15—C10	118.8 (3)

C5—C6—H6	121.8	C14—C15—H15	120.6
C7—C6—H6	121.8	C10—C15—H15	120.6
O1—C7—C6	125.1 (2)	C13—C16—H16A	109.5
O1—C7—C2	110.7 (2)	C13—C16—H16B	109.5
C6—C7—C2	124.1 (2)	H16A—C16—H16B	109.5
C1—C8—O1	110.8 (2)	C13—C16—H16C	109.5
C1—C8—C9	133.6 (2)	H16A—C16—H16C	109.5
O1—C8—C9	115.6 (2)	H16B—C16—H16C	109.5
C8—C9—H9A	109.5		
O2—S1—C1—C8	-126.3 (2)	C3—C2—C7—C6	-0.6 (4)
C10—S1—C1—C8	123.1 (2)	C1—C2—C7—C6	179.3 (2)
O2—S1—C1—C2	51.9 (3)	C2—C1—C8—O1	0.6 (3)
C10—S1—C1—C2	-58.7 (2)	S1—C1—C8—O1	179.22 (16)
C8—C1—C2—C3	179.7 (3)	C2—C1—C8—C9	179.3 (3)
S1—C1—C2—C3	1.3 (4)	S1—C1—C8—C9	-2.1 (4)
C8—C1—C2—C7	-0.2 (3)	C7—O1—C8—C1	-0.8 (3)
S1—C1—C2—C7	-178.6 (2)	C7—O1—C8—C9	-179.8 (2)
C7—C2—C3—C4	-0.8 (3)	O2—S1—C10—C11	1.7 (2)
C1—C2—C3—C4	179.4 (3)	C1—S1—C10—C11	114.7 (2)
C2—C3—C4—C5	1.1 (4)	O2—S1—C10—C15	176.9 (2)
C2—C3—C4—I1	-177.11 (17)	C1—S1—C10—C15	-70.1 (2)
O2 ⁱ —I1—C4—C3	159.7 (2)	C15—C10—C11—C12	1.4 (4)
O2 ⁱ —I1—C4—C5	-18.6 (5)	S1—C10—C11—C12	176.4 (2)
C3—C4—C5—C6	-0.2 (4)	C10—C11—C12—C13	0.1 (5)
I1—C4—C5—C6	178.08 (19)	C11—C12—C13—C14	-0.9 (5)
C4—C5—C6—C7	-1.1 (4)	C11—C12—C13—C16	178.7 (3)
C8—O1—C7—C6	-179.0 (2)	C12—C13—C14—C15	0.3 (4)
C8—O1—C7—C2	0.7 (3)	C16—C13—C14—C15	-179.3 (3)
C5—C6—C7—O1	-178.9 (2)	C13—C14—C15—C10	1.1 (4)
C5—C6—C7—C2	1.5 (4)	C11—C10—C15—C14	-2.0 (4)
C3—C2—C7—O1	179.8 (2)	S1—C10—C15—C14	-177.1 (2)
C1—C2—C7—O1	-0.3 (3)		

Symmetry code: (i) $x, -y+3/2, z+1/2$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C5—H5 \cdots O1 ⁱⁱ	0.95	2.52	3.459 (3)	170
C9—H9A \cdots O2 ⁱⁱⁱ	0.98	2.52	3.325 (3)	139
C15—H15 \cdots O2 ⁱⁱⁱ	0.95	2.58	3.499 (3)	162

Symmetry codes: (ii) $-x+2, y+1/2, -z+3/2$; (iii) $x, -y+1/2, z+1/2$.