

(Phenyliodosyl)benzene tosylate dihydrate

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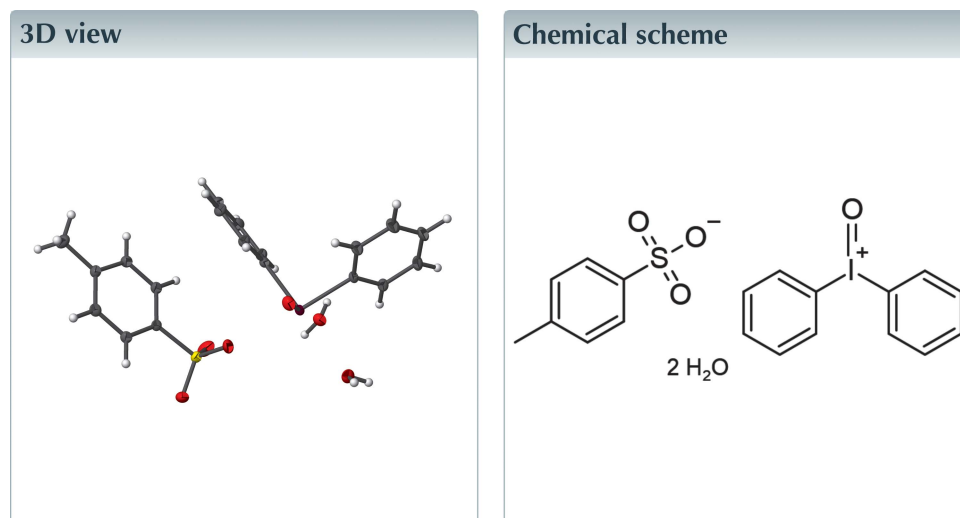
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The structure of the title salt (systematic name: oxodiphenyl- λ^5 -iodanylium 4-methylbenzenesulfonate dihydrate), $C_{12}H_{10}IO^+ \cdot C_7H_7O_3S^- \cdot 2H_2O$, at 150 K, has monoclinic ($P2_1/c$) symmetry. The molecular structure features an angular (phenyliodosyl)benzene cation, the geometry of which was hitherto undescribed in the literature: in the cation, both I—C bonds are approximately normal to the I=O bond, forming a C—I—C angle of $95.36(4)^\circ$. The crystal structure displays O—H \cdots O, O—H \cdots I and O—H \cdots S hydrogen bonding.



Structure description

The crystal structure of (phenyliodosyl)benzene tosylate dihydrate (**1**) is shown in Fig. 1. A partial packing structure is shown in Fig. 2. The title compound **1** crystallizes from water *via* slow cooling to 293 K to the monoclinic crystal system with space group $P2_1/c$. This iodosyl salt arose from the reaction of iodoxybenzene with sodium hydroxide (Fig. 3). The intermediate formed was captured with *p*-toluene sulfonic acid, generating **1**.

In compound **1** there are two phenyl rings connected to the iodine centre, C1—I1—C7, with a bond angle of $95.36(4)^\circ$. The bond lengths of C1—I1 and C7—I1 are 2.1289 (11) and 2.1370 (12) Å, respectively. These values are comparable to the sum of the van der Waals radii (2.05 Å; Bondi, 1964). These bond lengths are comparable to those found in 1,1,1-triacetoxy-1,1-dihydro-1,2-benziodoxol-3(1*H*)-one, *i.e.* the Dess–Martin periodinane, with a C—I bond length between the phenyl and iodine of 2.1025 (16) Å (Schröckeneder *et al.*, 2012). A secondary bonding interaction with the *p*-toluene sulfonate anion, O4 \cdots I1 [2.7076 (10) Å], resides nearly perpendicular at $77.55(4)^\circ$ to the I1—O1 bond. This bond length is shorter than the sum (3.05 Å) of the covalent radii, and this is analogous to secondary bonding that was observed by Rentzeperis in his bis(diphenyliodonium *I*-oxide) diacetate trihydrate, between the acetate anion and the iodo-

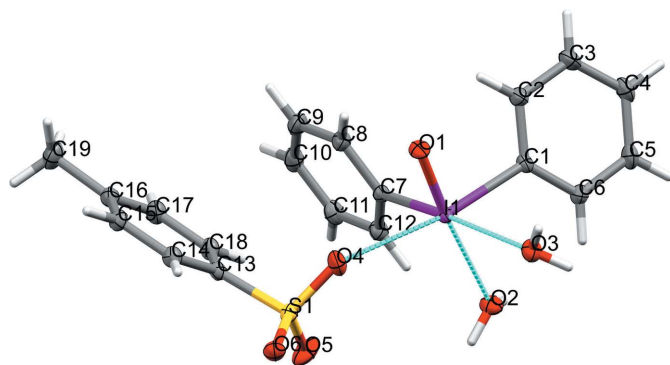


Figure 1
The title compound **1** with 50% displacement ellipsoids. All hydrogen labels are omitted for clarity.

nium *I*-oxide centre (Bozopoulos & Rentzeperis, 1987). Zhdankin found similar bond lengths of coordination between the trifluoromethanesulfonate oxygen anion and the cationic iodonium centre (2.797 Å) in [(arylsulfonyl)methyl](phenyl)iodonium trifluoromethanesulfonate (Zhdankin *et al.*, 1997). Additionally, Rentzeperis found similar coordinating distances with this acetate ion [2.449 (7) Å] and the oxygen atoms of the three water molecules, at 2.449 (7), 2.732 (9) and 2.732 (7) Å, respectively (Bozopoulos & Rentzeperis, 1987). In the title compound, the I1–O1 bond of 1.8108 (9) Å indicates double-bond character, as the computed double-bond length *via* van der Waals radii predicts 1.86 Å (Bondi, 1964). Rentzeperis found a similar bond length, 1.842 (6) Å, in bis(diphenyliodonium *I*-oxide) diacetate trihydrate (Bozopoulos & Rentzeperis, 1987).

Zhdankin synthesized a tosylate derivative of 2-iodoxybenzoic acid. Crystals of the final product could not be isolated, but the intermediate mixed tosylate-acetate derivative was analysed. The I–O bond lengths in this iodine(V) compound had distances of 2.080 (2), 2.213 (2), 2.027 (2) and 1.998 (2) Å (Yusubov *et al.*, 2013). These values are in accordance with a single-bond interaction of iodine with oxygen, further indicating that the I–O bond distance in **1** is of a double-bond nature. Additionally, bond lengths being shorter than predicted in hypervalent iodine compounds have been studied previously (Koser *et al.*, 1976). Koser confirmed the

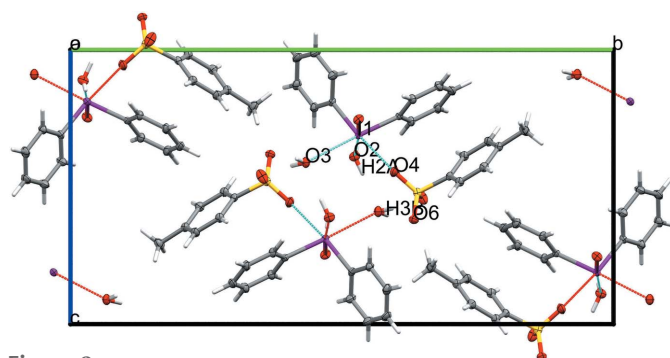


Figure 2
The unit cell as viewed down the *a* axis showing intermolecular hydrogen bonds as dashed lines.

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

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
O2–H2A···O3 ⁱ	0.86 (2)	1.92 (2)	2.7563 (14)	165 (2)
O2–H2B···I1 ⁱⁱ	0.85 (2)	3.14 (2)	3.9397 (10)	158.1 (18)
O2–H2B···O1 ⁱⁱ	0.85 (2)	1.91 (2)	2.7443 (14)	169 (2)
O3–H3A···S1 ⁱ	0.84 (2)	3.00 (2)	3.7847 (11)	155.7 (17)
O3–H3A···O5 ⁱ	0.84 (2)	1.92 (2)	2.7625 (15)	174.6 (19)
O3–H3B···S1 ⁱⁱⁱ	0.79 (2)	2.89 (2)	3.5812 (10)	147.5 (18)
O3–H3B···O6 ⁱⁱⁱ	0.79 (2)	1.99 (2)	2.7776 (14)	171 (2)

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

I–O single bond length was shorter (1.91 Å) than the computed distance (1.96 Å) in his seminal work on hydroxy(tosyloxy)iodobenzene. Additionally, in **1**, secondary coordination of the iodonium *I*-oxide centre with neighbouring water molecules indicates a close contact *via* the I1···O2 and I1···O3 with bond distances of 2.5674 (10) and 2.8118 (10) Å, respectively.

The title compound forms a distorted octahedral geometry in accordance with comparison to a VSEPR model. The O1–I1–O2 bond angle of 175.27 (4)°, the C7–I1–O3 angle of 176.33 (4)°, the O1–I1–O4 angle of 77.55 (4)° with the coordinating tosylate anion and the C1–I1–C7 angle of 95.36 (4)° complete the distorted octahedral geometry. The accompanying tosylate anion and water molecules occupy apical and equatorial positions to stabilize the monomeric complex. Bis(diphenyliodonium *I*-oxide) diacetate trihydrate also adopted a distorted octahedral geometry, albeit *via* a dimeric coordinating structure (Bozopoulos & Rentzeperis, 1987). In this complex, the asymmetric units form distorted trigonal-pyramidal arrangements, where the iodine atoms occupy the apices, resembling the IO₃[−] iodate anion. Secondary I···O interactions complete the distorted octahedral geometry around each individual iodine atom. The title complex **1** does not dimerize like the Rentzeperis compound, most likely due to the bulky nature of the coordinating tosylate anion, along with additional hydrogen bonding of the sulfone O atoms and water O atoms with neighbouring water molecules.

Examination of the molecular packing as illustrated in Fig. 2 shows O3ⁱ···H2A and O6ⁱⁱⁱ···H3B contacts, with O···H distances of 1.92 (2) and 1.99 (2) Å, respectively, as viewed down the *a* axis (Fig. 2 and Table 1). These two coordinations inhibit aggregation of the iodonium centres as seen in bis(diphenyliodonium *I*-oxide) diacetate trihydrate (Bozopoulos & Rentzeperis, 1987).

Synthesis and crystallization

(Phenyliodosyl)benzene tosylate dihydrate was synthesized according to a modified procedure by Chen (2007) and is illustrated in Fig. 3. Iodobenzene (2.04 g, 10 mmol) was added to a water solution (20 ml) of sodium metaperiodate (4.7 g, 22 mmol) with a small amount of toluene (0.3 ml) to minimize steam distillation. The reaction was heated to reflux for 18 h

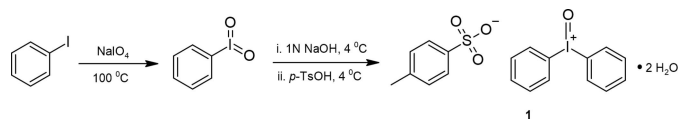


Figure 3
The synthetic route to obtain the title compound **1**.

and then cooled to room temperature. To the cooled reaction flask were added 50 ml of ice-cold water, and the white crystals that formed were filtered, washed with cold water (20 ml), cold chloroform (10 ml), and air-dried in a dark room until a constant weight was found (2.08 g, 8.81 mmol, 88% yield). The crude material was used in the next step without further purification. The iodoxybenzene (2.08 g, 8.81 mmol) was added to a stirred solution of 1 N NaOH (18.7 ml) pre-cooled to 277 K. The reaction was stirred for 1 h maintaining the temperature of the reaction below 281 K. The NaIO₃ that formed was filtered off. The filtrate was poured into a round-bottomed flask equipped with a magnetic stir bar and cooled to 277 K. With vigorous stirring *p*-toluene sulfonic acid monohydrate (2.87 g, 15.08 mmol) was added to the cooled filtrate and a white precipitate formed. The suspension was stirred for an additional 30 min and then filtered. The compound was washed with a minimal amount of diethyl ether (10 ml) and ice-cold water (10 ml). The product (1.75 g, 3.47 mmol, 47.5% yield) matched known ¹H, ¹³C and FTIR data (Chen, 2007). A sample for crystallographic analysis was prepared by dissolving the sample in a minimal amount of boiling water and allowing for slow cooling in an insulated thermal bath, insuring that the temperature took at least two days to return to room temperature. After additional cooling at room temperature for four days, the crystals that formed were suitable for X-ray analysis.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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Table 2
Experimental details.

Crystal data	
Chemical formula	C ₁₂ H ₁₀ IO ⁺ ·C ₇ H ₇ O ₃ S ⁻ ·2H ₂ O
<i>M_r</i>	504.32
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>c</i>
Temperature (K)	150
<i>a</i> , <i>b</i> , <i>c</i> (Å)	6.1823 (3), 24.9509 (11), 12.7606 (6)
β (°)	100.257 (2)
<i>V</i> (Å ³)	1936.92 (16)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	1.79
Crystal size (mm)	0.23 × 0.18 × 0.15
Data collection	
Diffractometer	Bruker AXS D8 Quest diffractometer with PhotonII charge-integrating pixel array detector (CPAD)
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2021)
<i>T_{min}</i> , <i>T_{max}</i>	0.644, 0.747
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	55248, 7429, 6815
<i>R_{int}</i>	0.033
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.772
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.018, 0.042, 1.09
No. of reflections	7429
No. of parameters	258
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	0.49, -0.39

Computer programs: *APEX4* and *SAINT* (Bruker, 2021), *SHELXT2018/2* (Sheldrick, 2015a), *SHELXL2018/3* (Sheldrick, 2015b), *Mercury* (Macrae *et al.*, 2020) and *publCIF* (Westrip, 2010).

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full crystallographic data

IUCrData (2022). 7, x220522 [https://doi.org/10.1107/S2414314622005223]

(Phenylidosyl)benzene tosylate dihydrate

Timothy J. Smith, Gerald Koser, Yi Chen, Matthias Zeller, Rocco Iacino and Nichole Selzer

Oxidiphenyl- λ^5 -iodanylium 4-methylbenzenesulfonate dihydrate*Crystal data*

$C_{12}H_{10}IO^+ \cdot C_7H_7O_3S^- \cdot 2H_2O$

$M_r = 504.32$

Monoclinic, $P2_1/c$

$a = 6.1823$ (3) Å

$b = 24.9509$ (11) Å

$c = 12.7606$ (6) Å

$\beta = 100.257$ (2)°

$V = 1936.92$ (16) Å³

$Z = 4$

$F(000) = 1008$

$D_x = 1.729$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9659 reflections

$\theta = 3.4$ – 33.2 °

$\mu = 1.79$ mm⁻¹

$T = 150$ K

Fragment, colourless

$0.23 \times 0.18 \times 0.15$ mm

Data collection

Bruker AXS D8 Quest
diffractometer with PhotonII charge-integrating
pixel array detector (CPAD)

Radiation source: fine focus sealed tube X-ray
source

Triumph curved graphite crystal
monochromator

Detector resolution: 7.4074 pixels mm⁻¹

ω and φ scans

Absorption correction: multi-scan
(SADABS; Bruker, 2021)

$T_{\min} = 0.644$, $T_{\max} = 0.747$

55248 measured reflections

7429 independent reflections

6815 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.033$

$\theta_{\max} = 33.3$ °, $\theta_{\min} = 2.9$ °

$h = -9 \rightarrow 9$

$k = -38 \rightarrow 38$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.018$

$wR(F^2) = 0.042$

$S = 1.09$

7429 reflections

258 parameters

0 restraints

Primary atom site location: dual

Secondary atom site location: difference Fourier
map

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0115P)^2 + 1.2666P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.004$

$\Delta\rho_{\max} = 0.49$ e Å⁻³

$\Delta\rho_{\min} = -0.39$ e Å⁻³

Extinction correction: SHELXL2018/3
(Sheldrick, 2015b),

$F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.00186 (16)

Special details

Refinement. H atoms attached to carbon were positioned geometrically and constrained to ride on their parent atoms. C—H bond distances were constrained to 0.95 Å for aromatic CH moieties, and to 0.98 Å for the CH₃ group, respectively. Water H atom positions were freely refined. $U_{\text{iso}}(\text{H})$ values were set to a multiple of $U_{\text{eq}}(\text{carrier C/O})$, with 1.5 for CH₃ and OH, and 1.2 for CH units, respectively.

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	0.68788 (2)	0.46909 (2)	0.68867 (2)	0.01154 (3)
S1	0.78701 (5)	0.36098 (2)	0.47914 (2)	0.01522 (5)
O1	0.97764 (14)	0.46950 (4)	0.74614 (8)	0.01780 (17)
O2	0.27627 (16)	0.47673 (4)	0.61133 (8)	0.01981 (18)
H2A	0.242 (3)	0.4622 (8)	0.5495 (17)	0.030*
H2B	0.173 (3)	0.4724 (8)	0.6458 (17)	0.030*
O3	0.75786 (17)	0.56665 (4)	0.58959 (8)	0.01987 (18)
H3A	0.665 (3)	0.5916 (8)	0.5781 (16)	0.030*
H3B	0.869 (4)	0.5829 (8)	0.6024 (16)	0.030*
O4	0.86090 (18)	0.40372 (4)	0.55568 (8)	0.02297 (19)
O5	0.55011 (16)	0.35301 (5)	0.46211 (10)	0.0294 (2)
O6	0.87304 (16)	0.36783 (4)	0.38086 (7)	0.02007 (18)
C1	0.5945 (2)	0.51811 (5)	0.80971 (9)	0.0142 (2)
C2	0.7369 (2)	0.51635 (6)	0.90699 (11)	0.0212 (2)
H2	0.862288	0.493668	0.917364	0.025*
C3	0.6902 (3)	0.54883 (6)	0.98885 (11)	0.0249 (3)
H3	0.783620	0.548237	1.056562	0.030*
C4	0.5077 (3)	0.58201 (6)	0.97162 (11)	0.0236 (3)
H4	0.477050	0.604223	1.027645	0.028*
C5	0.3694 (2)	0.58310 (6)	0.87349 (12)	0.0229 (3)
H5	0.244730	0.606019	0.862871	0.027*
C6	0.4115 (2)	0.55088 (5)	0.79013 (10)	0.0182 (2)
H6	0.317967	0.551429	0.722456	0.022*
C7	0.6142 (2)	0.39479 (5)	0.75784 (9)	0.0139 (2)
C8	0.7930 (2)	0.37027 (5)	0.82033 (10)	0.0187 (2)
H8	0.934473	0.386350	0.830739	0.022*
C9	0.7592 (3)	0.32126 (6)	0.86755 (11)	0.0235 (3)
H9	0.878788	0.303693	0.911211	0.028*
C10	0.5521 (3)	0.29802 (5)	0.85114 (11)	0.0231 (3)
H10	0.530416	0.264622	0.883500	0.028*
C11	0.3762 (2)	0.32346 (5)	0.78748 (11)	0.0217 (2)
H11	0.234867	0.307259	0.776581	0.026*
C12	0.4048 (2)	0.37264 (5)	0.73933 (10)	0.0178 (2)
H12	0.285534	0.390249	0.695560	0.021*
C13	0.90551 (19)	0.30148 (5)	0.53950 (9)	0.0143 (2)
C14	1.1068 (2)	0.28329 (5)	0.51879 (10)	0.0166 (2)
H14	1.177697	0.301415	0.468824	0.020*
C15	1.2027 (2)	0.23823 (5)	0.57228 (10)	0.0180 (2)
H15	1.339293	0.225549	0.557827	0.022*

C16	1.1023 (2)	0.21125 (5)	0.64677 (10)	0.0180 (2)
C17	0.8999 (2)	0.23004 (5)	0.66564 (11)	0.0195 (2)
H17	0.828525	0.211958	0.715483	0.023*
C18	0.8014 (2)	0.27479 (5)	0.61256 (10)	0.0172 (2)
H18	0.663586	0.287143	0.626024	0.021*
C19	1.2112 (3)	0.16377 (6)	0.70690 (13)	0.0283 (3)
H19A	1.137702	0.155730	0.767074	0.042*
H19B	1.366266	0.171982	0.733455	0.042*
H19C	1.200511	0.132671	0.659362	0.042*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
II	0.00981 (3)	0.01211 (4)	0.01291 (4)	-0.00066 (2)	0.00260 (2)	-0.00161 (2)
S1	0.01308 (12)	0.01470 (12)	0.01821 (13)	0.00026 (10)	0.00368 (10)	0.00096 (10)
O1	0.0086 (3)	0.0232 (4)	0.0211 (4)	-0.0009 (3)	0.0013 (3)	-0.0040 (3)
O2	0.0141 (4)	0.0254 (5)	0.0199 (4)	-0.0026 (4)	0.0030 (3)	-0.0025 (4)
O3	0.0179 (4)	0.0171 (4)	0.0250 (5)	-0.0021 (4)	0.0049 (4)	0.0001 (3)
O4	0.0306 (5)	0.0158 (4)	0.0245 (5)	0.0005 (4)	0.0104 (4)	-0.0040 (4)
O5	0.0127 (4)	0.0304 (6)	0.0440 (6)	0.0006 (4)	0.0024 (4)	0.0130 (5)
O6	0.0234 (4)	0.0212 (4)	0.0160 (4)	0.0004 (4)	0.0045 (3)	0.0021 (3)
C1	0.0147 (5)	0.0131 (5)	0.0153 (5)	-0.0004 (4)	0.0040 (4)	-0.0032 (4)
C2	0.0196 (6)	0.0242 (6)	0.0186 (6)	0.0043 (5)	0.0000 (4)	-0.0052 (5)
C3	0.0286 (7)	0.0280 (7)	0.0170 (6)	0.0019 (6)	0.0013 (5)	-0.0069 (5)
C4	0.0305 (7)	0.0209 (6)	0.0216 (6)	0.0003 (5)	0.0108 (5)	-0.0067 (5)
C5	0.0238 (6)	0.0192 (6)	0.0270 (6)	0.0055 (5)	0.0080 (5)	-0.0035 (5)
C6	0.0177 (5)	0.0174 (5)	0.0192 (5)	0.0029 (4)	0.0026 (4)	-0.0014 (4)
C7	0.0162 (5)	0.0127 (5)	0.0132 (5)	0.0003 (4)	0.0036 (4)	-0.0007 (4)
C8	0.0184 (5)	0.0180 (5)	0.0194 (6)	0.0027 (4)	0.0027 (4)	0.0006 (4)
C9	0.0288 (7)	0.0201 (6)	0.0214 (6)	0.0073 (5)	0.0037 (5)	0.0040 (5)
C10	0.0350 (7)	0.0162 (5)	0.0204 (6)	0.0015 (5)	0.0110 (5)	0.0022 (4)
C11	0.0252 (6)	0.0170 (6)	0.0242 (6)	-0.0047 (5)	0.0084 (5)	-0.0009 (5)
C12	0.0174 (5)	0.0161 (5)	0.0199 (5)	-0.0015 (4)	0.0033 (4)	0.0000 (4)
C13	0.0140 (5)	0.0130 (5)	0.0160 (5)	-0.0011 (4)	0.0028 (4)	-0.0010 (4)
C14	0.0152 (5)	0.0169 (5)	0.0187 (5)	-0.0009 (4)	0.0055 (4)	0.0002 (4)
C15	0.0150 (5)	0.0193 (6)	0.0201 (6)	0.0022 (4)	0.0046 (4)	-0.0005 (4)
C16	0.0208 (6)	0.0152 (5)	0.0181 (5)	0.0013 (4)	0.0037 (4)	-0.0003 (4)
C17	0.0225 (6)	0.0170 (5)	0.0208 (6)	-0.0004 (5)	0.0092 (5)	0.0010 (4)
C18	0.0159 (5)	0.0168 (5)	0.0203 (5)	-0.0001 (4)	0.0069 (4)	0.0001 (4)
C19	0.0334 (8)	0.0234 (7)	0.0292 (7)	0.0094 (6)	0.0090 (6)	0.0083 (5)

Geometric parameters (Å, °)

II—O1	1.8108 (9)	C7—C8	1.3849 (17)
II—C1	2.1289 (11)	C7—C12	1.3883 (17)
II—C7	2.1370 (12)	C8—C9	1.3954 (19)
II—O2	2.5674 (10)	C8—H8	0.9500
II—O4	2.7076 (10)	C9—C10	1.387 (2)

I1—O3	2.8118 (10)	C9—H9	0.9500
S1—O5	1.4555 (10)	C10—C11	1.390 (2)
S1—O6	1.4568 (10)	C10—H10	0.9500
S1—O4	1.4631 (10)	C11—C12	1.3975 (18)
S1—C13	1.7702 (12)	C11—H11	0.9500
O2—H2A	0.86 (2)	C12—H12	0.9500
O2—H2B	0.85 (2)	C13—C18	1.3931 (17)
O3—H3A	0.84 (2)	C13—C14	1.3935 (17)
O3—H3B	0.79 (2)	C14—C15	1.3919 (18)
C1—C6	1.3819 (17)	C14—H14	0.9500
C1—C2	1.3888 (18)	C15—C16	1.3973 (18)
C2—C3	1.3923 (19)	C15—H15	0.9500
C2—H2	0.9500	C16—C17	1.3970 (18)
C3—C4	1.385 (2)	C16—C19	1.5037 (19)
C3—H3	0.9500	C17—C18	1.3888 (18)
C4—C5	1.385 (2)	C17—H17	0.9500
C4—H4	0.9500	C18—H18	0.9500
C5—C6	1.3942 (18)	C19—H19A	0.9800
C5—H5	0.9500	C19—H19B	0.9800
C6—H6	0.9500	C19—H19C	0.9800
O1—I1—C1	94.57 (4)	C1—C6—H6	121.3
O1—I1—C7	96.08 (5)	C5—C6—H6	121.3
C1—I1—C7	95.36 (4)	C8—C7—C12	123.12 (12)
O1—I1—O2	175.27 (4)	C8—C7—I1	114.46 (9)
C1—I1—O2	81.83 (4)	C12—C7—I1	122.41 (9)
C7—I1—O2	87.34 (4)	C7—C8—C9	118.05 (12)
O1—I1—O4	77.55 (4)	C7—C8—H8	121.0
C1—I1—O4	171.59 (4)	C9—C8—H8	121.0
C7—I1—O4	82.72 (4)	C10—C9—C8	120.41 (13)
O2—I1—O4	106.19 (3)	C10—C9—H9	119.8
O1—I1—O3	87.53 (4)	C8—C9—H9	119.8
C1—I1—O3	84.96 (4)	C9—C10—C11	120.17 (13)
C7—I1—O3	176.33 (4)	C9—C10—H10	119.9
O2—I1—O3	89.09 (3)	C11—C10—H10	119.9
O4—I1—O3	97.47 (3)	C10—C11—C12	120.71 (13)
O5—S1—O6	113.54 (7)	C10—C11—H11	119.6
O5—S1—O4	112.68 (7)	C12—C11—H11	119.6
O6—S1—O4	111.76 (6)	C7—C12—C11	117.53 (12)
O5—S1—C13	106.12 (6)	C7—C12—H12	121.2
O6—S1—C13	106.66 (6)	C11—C12—H12	121.2
O4—S1—C13	105.37 (6)	C18—C13—C14	120.40 (11)
I1—O2—H2A	112.9 (14)	C18—C13—S1	119.17 (9)
I1—O2—H2B	125.5 (14)	C14—C13—S1	120.33 (9)
H2A—O2—H2B	110 (2)	C15—C14—C13	119.13 (11)
I1—O3—H3A	124.2 (14)	C15—C14—H14	120.4
I1—O3—H3B	123.4 (15)	C13—C14—H14	120.4
H3A—O3—H3B	101.5 (19)	C14—C15—C16	121.38 (12)

S1—O4—I1	138.26 (6)	C14—C15—H15	119.3
C6—C1—C2	123.26 (11)	C16—C15—H15	119.3
C6—C1—I1	121.86 (9)	C17—C16—C15	118.41 (12)
C2—C1—I1	114.81 (9)	C17—C16—C19	120.65 (12)
C1—C2—C3	117.98 (13)	C15—C16—C19	120.93 (12)
C1—C2—H2	121.0	C18—C17—C16	120.94 (12)
C3—C2—H2	121.0	C18—C17—H17	119.5
C4—C3—C2	120.03 (13)	C16—C17—H17	119.5
C4—C3—H3	120.0	C17—C18—C13	119.73 (12)
C2—C3—H3	120.0	C17—C18—H18	120.1
C5—C4—C3	120.63 (12)	C13—C18—H18	120.1
C5—C4—H4	119.7	C16—C19—H19A	109.5
C3—C4—H4	119.7	C16—C19—H19B	109.5
C4—C5—C6	120.63 (13)	H19A—C19—H19B	109.5
C4—C5—H5	119.7	C16—C19—H19C	109.5
C6—C5—H5	119.7	H19A—C19—H19C	109.5
C1—C6—C5	117.46 (12)	H19B—C19—H19C	109.5
O5—S1—O4—I1	5.82 (11)	C10—C11—C12—C7	-0.16 (19)
O6—S1—O4—I1	135.08 (8)	O5—S1—C13—C18	-36.61 (12)
C13—S1—O4—I1	-109.46 (9)	O6—S1—C13—C18	-157.97 (10)
C6—C1—C2—C3	0.8 (2)	O4—S1—C13—C18	83.11 (11)
I1—C1—C2—C3	177.82 (11)	O5—S1—C13—C14	147.06 (11)
C1—C2—C3—C4	-0.7 (2)	O6—S1—C13—C14	25.70 (12)
C2—C3—C4—C5	0.3 (2)	O4—S1—C13—C14	-93.22 (11)
C3—C4—C5—C6	-0.1 (2)	C18—C13—C14—C15	-0.18 (19)
C2—C1—C6—C5	-0.6 (2)	S1—C13—C14—C15	176.11 (10)
I1—C1—C6—C5	-177.39 (10)	C13—C14—C15—C16	-0.6 (2)
C4—C5—C6—C1	0.2 (2)	C14—C15—C16—C17	1.0 (2)
C12—C7—C8—C9	-0.70 (19)	C14—C15—C16—C19	-177.83 (13)
I1—C7—C8—C9	-179.34 (10)	C15—C16—C17—C18	-0.7 (2)
C7—C8—C9—C10	0.4 (2)	C19—C16—C17—C18	178.17 (13)
C8—C9—C10—C11	-0.1 (2)	C16—C17—C18—C13	-0.1 (2)
C9—C10—C11—C12	-0.1 (2)	C14—C13—C18—C17	0.51 (19)
C8—C7—C12—C11	0.56 (19)	S1—C13—C18—C17	-175.82 (10)
I1—C7—C12—C11	179.10 (9)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O2—H2 <i>A</i> ...O3 ⁱ	0.86 (2)	1.92 (2)	2.7563 (14)	165 (2)
O2—H2 <i>B</i> ...I1 ⁱⁱ	0.85 (2)	3.14 (2)	3.9397 (10)	158.1 (18)
O2—H2 <i>B</i> ...O1 ⁱⁱ	0.85 (2)	1.91 (2)	2.7443 (14)	169 (2)
O3—H3 <i>A</i> ...S1 ⁱ	0.84 (2)	3.00 (2)	3.7847 (11)	155.7 (17)
O3—H3 <i>A</i> ...O5 ⁱ	0.84 (2)	1.92 (2)	2.7625 (15)	174.6 (19)
O3—H3 <i>B</i> ...S1 ⁱⁱⁱ	0.79 (2)	2.89 (2)	3.5812 (10)	147.5 (18)
O3—H3 <i>B</i> ...O6 ⁱⁱⁱ	0.79 (2)	1.99 (2)	2.7776 (14)	171 (2)
C2—H2...O1	0.95	2.49	2.9805 (16)	112

C6—H6···O2	0.95	2.33	2.9408 (17)	122
C6—H6···O6 ⁱ	0.95	2.58	3.2562 (16)	129
C8—H8···O1	0.95	2.38	2.9521 (17)	119
C12—H12···O2	0.95	2.41	3.0957 (17)	129
C14—H14···O5 ^{iv}	0.95	2.65	3.4301 (16)	139

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