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rac-2-Iodo-3,4-dihydronaphthalen-1(2*H*)-one

Abdul Rauf Raza,[a] M. Nawaz Tahir,[b]* Ayesha Sultan,[a] Muhammad Danish[a] and Muhammad Sohail[a]

[a]Department of Chemistry, University of Sargodha, Sargodha, Pakistan, and [b]Department of Physics, University of Sargodha, Sargodha, Pakistan
Correspondence e-mail: dmntahir_uos@yahoo.com

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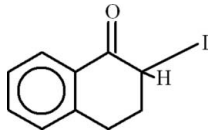
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; disorder in main residue; R factor = 0.024; wR factor = 0.059; data-to-parameter ratio = 18.5.

In the title compound, $C_{10}H_9IO$, the asymmetric unit contains two molecules, in which the iodo-bearing six-membered rings adopt envelope conformations [displacements of the flap atoms = 0.419 (3) and 0.431 (3) Å]. In both molecules, the I atoms are disordered over two set of sites in 0.54 (4):0.46 (4) and 0.71 (3):0.29 (3) ratios. In the crystal, the packing features a weak C—H$\cdots\pi$ interaction.

Related literature

For a related structure, see: Haddad (1986).



Experimental

Crystal data

$C_{10}H_9IO$
M_r = 272.07
Monoclinic, $P2_1/c$
a = 6.115 (5) Å
b = 19.658 (4) Å
c = 15.896 (5) Å
β = 90.551 (5)°
V = 1910.7 (17) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 3.31 mm^{-1}
T = 296 K
0.28 × 0.20 × 0.18 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.685, T_{max} = 0.717
19043 measured reflections
4397 independent reflections
3632 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.059
S = 1.07
4397 reflections
238 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C5—H5$\cdots Cg3$	0.93	2.95	3.700 (4)	139

*Cg*3 is the centroid of the C11–C16 ring.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); 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 *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5236).

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supporting information

Acta Cryst. (2009). E**65**, o3172 [doi:10.1107/S1600536809049095]

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Abdul Rauf Raza, M. Nawaz Tahir, Ayesha Sultan, Muhammad Danish and Muhammad Sohail

S1. Comment

The title compound (I, Fig. 1) is an intermediate for the total synthesis of steroidal hormones. The ctystal structures of (II) 2,2-Dibromo-3,4-dihydro-1(2*H*)-naphthalenone (Haddad, 1986) has been published, which seems relavent to (I).

The asymmetric unit of title compound consists of two individual molecules which are clearly racemate. In the molecule having (*S*)-configuration, the I-atom containing ring A (C1/C6/C7—C10) is twisted with maximum puckering amplitude Q_T = 0.431 (3) Å, whereas in (*R*)-configuration the puckering parameter is Q_T = 0.419 (3) Å. In two molecules the groups of benzene rings along with two adjacent C-atoms, C (C1—C6/C7/C10) and D (C11—C16/C17/C20) are planar with maximum r. m. s. deviations of 0.0114 and 0.0280 Å respectively, from the respective mean square planes. The dihedral angle between C/D is 66.83 (7) Å. In the first molecule the I-atom is disordered over two set of sites having occupancy ratio of 0.54 (4):0.46 (4). Similarly in the other molecule the I-atom is disordered over two set of sites having occupancy ratio of 0.71 (3):0.29 (3). The molecules are stabilized due to C–H···π interactions (Table 1).

S2. Experimental

A solution of I_2 (7.75 g, 30.5 mmol) in $CHCl_3$ was added as drops to a solution of 1-tetralone (2.198 g, 15.2 mmol) in acetic acid (9.156 g, 0.1526 mol) and refluxed for 28 h. The H_2O (30 ml) was added for partitioning. The reaction mixture was extracted with $CHCl_3$ (3 × 15 ml). The combined organic layer was concentrated *in vacuo*, the crude was dissolved in ethyl acetate, washed with 5% $Na_2S_2O_3$ (2 × 15 ml), dried over anhydrous Na_2SO_4, filtered, boiled with charcoal, concentrated under reduce pressure and allowed for crystallization, which afforded colourless prisms (89%) of (I).

S3. Refinement

The other H-atoms were positioned geometrically (C–H = 0.93–0.98 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C).

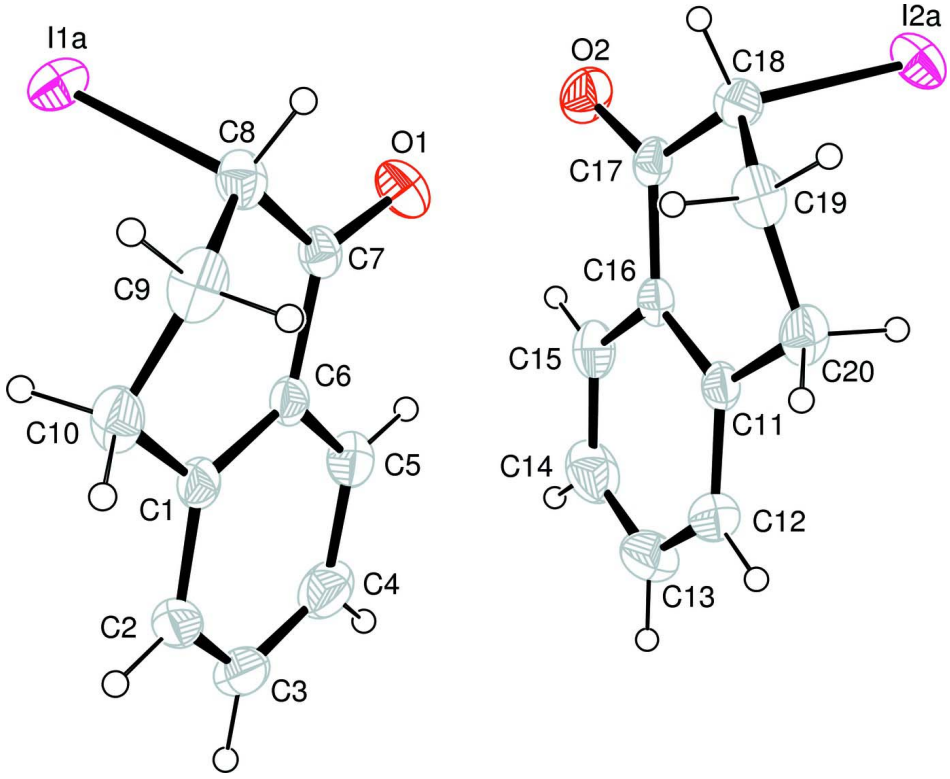

Figure 1

View of (I) with the I atoms having greater occupancies. The displacement ellipsoids are drawn at the 30% probability level.

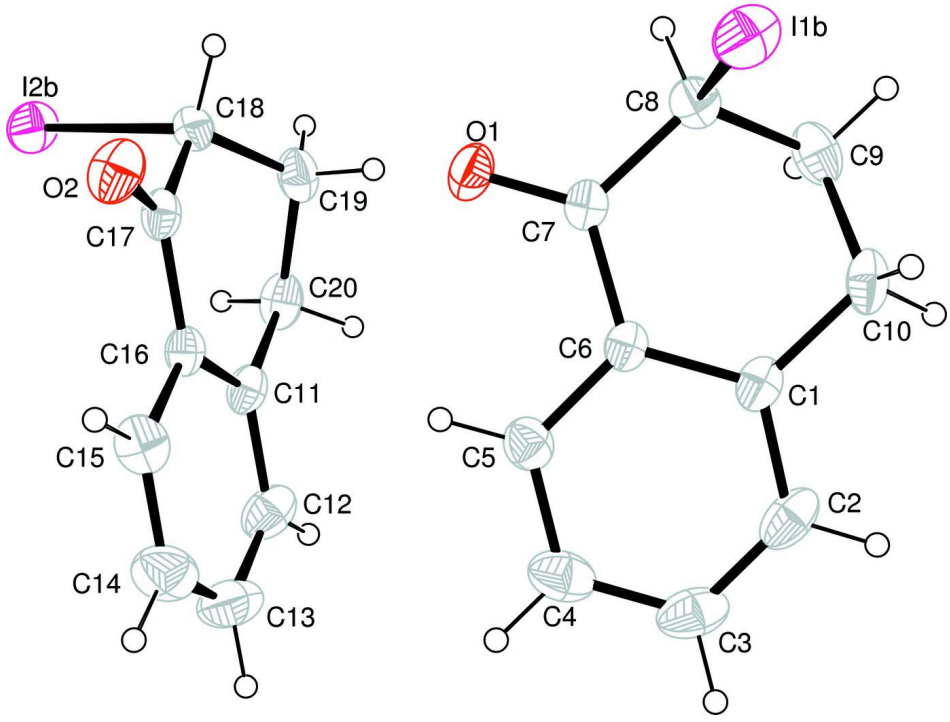

Figure 2

View of (I) with the I atoms having lesser occupancies. The displacement ellipsoids are drawn at the 30% probability level.

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Crystal data

$C_{10}H_9IO$
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$c = 15.896\ (5)$ Å
$\beta = 90.551\ (5)°$
$V = 1910.7\ (17)$ Å^3
$Z = 8$
$F(000) = 1040$
$D_x = 1.892$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4397 reflections
$\theta = 2.4–27.8°$
$\mu = 3.31$ mm^{-1}
$T = 296$ K
Prism, colourless
0.28 × 0.20 × 0.18 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 7.50 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.685$, $T_{max} = 0.717$
19043 measured reflections
4397 independent reflections
3632 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 27.8°$, $\theta_{min} = 2.4°$
$h = -8\rightarrow7$
$k = -25\rightarrow24$
$l = -20\rightarrow20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.059$
$S = 1.07$
4397 reflections
238 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
$w = 1/[\sigma^2(F_o^2) + (0.0273P)^2 + 0.6672P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.50$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00283 (17)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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 $F^2 > \sigma(F^2)$ 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}	Occ. (<1)
I1A	0.8199 (5)	0.0828 (2)	0.36895 (19)	0.0502 (6)	0.54 (4)
I1B	0.8299 (7)	0.0783 (3)	0.3658 (2)	0.0622 (8)	0.46 (4)
O1	0.6627 (3)	0.25061 (10)	0.31382 (12)	0.0512 (6)	
C1	0.9910 (4)	0.17057 (12)	0.15222 (17)	0.0408 (8)	
C2	0.9965 (5)	0.16572 (14)	0.06450 (19)	0.0573 (10)	
C3	0.8386 (6)	0.19473 (15)	0.01454 (19)	0.0637 (10)	
C4	0.6694 (6)	0.23029 (15)	0.05043 (19)	0.0621 (11)	
C5	0.6596 (4)	0.23677 (13)	0.13679 (17)	0.0473 (9)	
C6	0.8181 (3)	0.20662 (11)	0.18883 (15)	0.0350 (7)	
C7	0.8027 (3)	0.21515 (11)	0.28175 (15)	0.0356 (7)	
C8	0.9707 (4)	0.17888 (13)	0.33563 (17)	0.0439 (8)	
C9	1.1881 (4)	0.16933 (15)	0.2919 (2)	0.0546 (9)	
C10	1.1619 (4)	0.13632 (14)	0.20632 (19)	0.0518 (9)	
I2A	0.2833 (4)	0.51127 (14)	0.4019 (2)	0.0524 (4)	0.71 (3)
I2B	0.2948 (9)	0.5150 (3)	0.3949 (5)	0.0551 (13)	0.29 (3)
O2	0.1573 (3)	0.33920 (10)	0.34826 (14)	0.0590 (7)	
C11	0.4861 (4)	0.42382 (11)	0.19085 (17)	0.0395 (8)	
C12	0.4975 (5)	0.42831 (14)	0.1038 (2)	0.0593 (11)	
C13	0.3390 (7)	0.39932 (18)	0.0525 (2)	0.0735 (13)	
C14	0.1666 (6)	0.36502 (18)	0.0870 (2)	0.0725 (12)	
C15	0.1531 (4)	0.35857 (13)	0.1724 (2)	0.0528 (10)	
C16	0.3121 (3)	0.38740 (11)	0.22589 (16)	0.0366 (7)	
C17	0.2928 (4)	0.37754 (11)	0.31794 (16)	0.0385 (7)	
C18	0.4509 (4)	0.41620 (12)	0.37374 (17)	0.0431 (8)	

C19	0.6704 (4)	0.42740 (14)	0.33282 (19)	0.0504 (9)
C20	0.6513 (4)	0.45891 (14)	0.24640 (19)	0.0496 (9)
H2	1.11050	0.14215	0.03943	0.0687*
H3	0.84562	0.19042	−0.04364	0.0763*
H4	0.56189	0.24993	0.01653	0.0746*
H5	0.54622	0.26144	0.16068	0.0568*
H8	0.99519	0.20536	0.38710	0.0527*
H9A	1.25810	0.21327	0.28537	0.0654*
H9B	1.28268	0.14126	0.32686	0.0654*
H10A	1.30098	0.13765	0.17757	0.0620*
H10B	1.12210	0.08895	0.21380	0.0620*
H12	0.61398	0.45126	0.07953	0.0712*
H13	0.34957	0.40313	−0.00567	0.0882*
H14	0.05908	0.34619	0.05236	0.0868*
H15	0.03679	0.33471	0.19547	0.0632*
H18	0.47290	0.39074	0.42614	0.0517*
H19A	0.75860	0.45680	0.36852	0.0605*
H19B	0.74537	0.38409	0.32833	0.0605*
H20A	0.61044	0.50634	0.25223	0.0596*
H20B	0.79302	0.45732	0.21957	0.0596*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1A	0.0533 (13)	0.0521 (9)	0.0453 (9)	−0.0069 (5)	0.0019 (5)	0.0139 (8)
I1B	0.084 (2)	0.0428 (9)	0.0599 (13)	−0.0067 (6)	0.0130 (10)	−0.0018 (11)
O1	0.0456 (10)	0.0531 (11)	0.0550 (12)	0.0073 (8)	0.0063 (8)	−0.0161 (9)
C1	0.0450 (12)	0.0273 (12)	0.0504 (16)	−0.0049 (9)	0.0138 (11)	0.0004 (11)
C2	0.0773 (19)	0.0391 (15)	0.0561 (19)	−0.0096 (13)	0.0319 (16)	−0.0049 (13)
C3	0.102 (2)	0.0490 (17)	0.0402 (16)	−0.0163 (17)	0.0091 (16)	0.0025 (14)
C4	0.083 (2)	0.0521 (18)	0.0510 (19)	−0.0057 (15)	−0.0130 (16)	0.0118 (14)
C5	0.0502 (14)	0.0387 (14)	0.0530 (17)	0.0023 (11)	−0.0036 (12)	0.0000 (12)
C6	0.0370 (11)	0.0243 (11)	0.0439 (14)	−0.0035 (9)	0.0054 (10)	−0.0011 (10)
C7	0.0322 (11)	0.0297 (12)	0.0448 (14)	−0.0045 (9)	0.0018 (10)	−0.0046 (10)
C8	0.0442 (13)	0.0389 (14)	0.0485 (15)	−0.0046 (10)	−0.0082 (11)	−0.0064 (11)
C9	0.0346 (12)	0.0499 (16)	0.079 (2)	−0.0019 (11)	−0.0078 (12)	0.0078 (15)
C10	0.0379 (13)	0.0419 (15)	0.076 (2)	0.0064 (10)	0.0160 (13)	0.0039 (14)
I2A	0.0475 (8)	0.0585 (9)	0.0512 (5)	0.0129 (3)	−0.0063 (3)	−0.0177 (5)
I2B	0.077 (3)	0.0322 (19)	0.0562 (13)	0.0131 (8)	0.0055 (13)	−0.0043 (12)
O2	0.0550 (11)	0.0514 (12)	0.0709 (14)	−0.0111 (9)	0.0206 (10)	0.0053 (10)
C11	0.0416 (12)	0.0274 (12)	0.0496 (16)	0.0011 (9)	0.0093 (11)	−0.0039 (10)
C12	0.0745 (19)	0.0488 (17)	0.0551 (19)	−0.0020 (14)	0.0204 (15)	0.0010 (14)
C13	0.105 (3)	0.071 (2)	0.0446 (18)	0.006 (2)	0.0004 (18)	−0.0078 (16)
C14	0.086 (2)	0.064 (2)	0.067 (2)	−0.0004 (18)	−0.0220 (19)	−0.0170 (17)
C15	0.0511 (15)	0.0381 (14)	0.069 (2)	−0.0081 (11)	−0.0073 (13)	−0.0075 (13)
C16	0.0355 (11)	0.0259 (12)	0.0484 (15)	0.0012 (9)	0.0025 (10)	−0.0033 (10)
C17	0.0356 (11)	0.0283 (12)	0.0517 (15)	0.0032 (9)	0.0071 (10)	0.0012 (10)
C18	0.0445 (13)	0.0427 (14)	0.0420 (15)	0.0102 (10)	−0.0023 (11)	0.0008 (11)

C19	0.0341 (12)	0.0504 (16)	0.0665 (19)	0.0033 (10)	−0.0068 (12)	−0.0087 (13)
C20	0.0347 (12)	0.0433 (15)	0.071 (2)	−0.0092 (10)	0.0103 (12)	−0.0039 (13)

Geometric parameters (Å, º)

I1A—C8	2.170 (5)	C9—H9B	0.9700
I1B—C8	2.211 (6)	C10—H10A	0.9700
I2A—C18	2.180 (4)	C10—H10B	0.9700
I2B—C18	2.192 (7)	C11—C12	1.389 (4)
O1—C7	1.220 (3)	C11—C16	1.402 (3)
O2—C17	1.223 (3)	C11—C20	1.503 (4)
C1—C6	1.404 (3)	C12—C13	1.384 (5)
C1—C2	1.398 (4)	C13—C14	1.370 (5)
C1—C10	1.506 (4)	C14—C15	1.367 (5)
C2—C3	1.369 (5)	C15—C16	1.405 (4)
C3—C4	1.377 (5)	C16—C17	1.482 (4)
C4—C5	1.381 (4)	C17—C18	1.511 (4)
C5—C6	1.400 (4)	C18—C19	1.513 (4)
C6—C7	1.490 (4)	C19—C20	1.510 (4)
C7—C8	1.510 (4)	C12—H12	0.9300
C8—C9	1.518 (4)	C13—H13	0.9300
C9—C10	1.514 (4)	C14—H14	0.9300
C2—H2	0.9300	C15—H15	0.9300
C3—H3	0.9300	C18—H18	0.9800
C4—H4	0.9300	C19—H19A	0.9700
C5—H5	0.9300	C19—H19B	0.9700
C8—H8	0.9800	C20—H20A	0.9700
C9—H9A	0.9700	C20—H20B	0.9700
C2—C1—C6	118.2 (2)	C12—C11—C16	118.3 (2)
C2—C1—C10	121.1 (2)	C12—C11—C20	121.1 (2)
C6—C1—C10	120.7 (2)	C16—C11—C20	120.6 (2)
C1—C2—C3	121.8 (3)	C11—C12—C13	121.3 (3)
C2—C3—C4	120.0 (3)	C12—C13—C14	120.3 (3)
C3—C4—C5	119.9 (3)	C13—C14—C15	119.8 (3)
C4—C5—C6	120.8 (2)	C14—C15—C16	121.0 (3)
C1—C6—C5	119.3 (2)	C11—C16—C15	119.3 (2)
C1—C6—C7	121.5 (2)	C11—C16—C17	121.8 (2)
C5—C6—C7	119.21 (19)	C15—C16—C17	118.9 (2)
O1—C7—C6	122.0 (2)	O2—C17—C16	122.1 (2)
O1—C7—C8	120.6 (2)	O2—C17—C18	120.7 (2)
C6—C7—C8	117.38 (18)	C16—C17—C18	117.2 (2)
I1A—C8—C7	105.13 (17)	I2A—C18—C17	104.62 (17)
I1A—C8—C9	112.4 (2)	I2A—C18—C19	112.60 (18)
I1B—C8—C7	106.32 (18)	I2B—C18—C17	105.0 (2)
I1B—C8—C9	109.5 (2)	I2B—C18—C19	109.1 (2)
C7—C8—C9	113.1 (2)	C17—C18—C19	112.7 (2)
C8—C9—C10	112.3 (2)	C18—C19—C20	112.9 (2)

C1—C10—C9	112.9 (2)	C11—C20—C19	113.1 (2)
C1—C2—H2	119.00	C11—C12—H12	119.00
C3—C2—H2	119.00	C13—C12—H12	119.00
C2—C3—H3	120.00	C12—C13—H13	120.00
C4—C3—H3	120.00	C14—C13—H13	120.00
C3—C4—H4	120.00	C13—C14—H14	120.00
C5—C4—H4	120.00	C15—C14—H14	120.00
C4—C5—H5	120.00	C14—C15—H15	119.00
C6—C5—H5	120.00	C16—C15—H15	119.00
I1A—C8—H8	109.00	I2A—C18—H18	109.00
I1B—C8—H8	111.00	I2B—C18—H18	112.00
C7—C8—H8	109.00	C17—C18—H18	109.00
C9—C8—H8	109.00	C19—C18—H18	109.00
C8—C9—H9A	109.00	C18—C19—H19A	109.00
C8—C9—H9B	109.00	C18—C19—H19B	109.00
C10—C9—H9A	109.00	C20—C19—H19A	109.00
C10—C9—H9B	109.00	C20—C19—H19B	109.00
H9A—C9—H9B	108.00	H19A—C19—H19B	108.00
C1—C10—H10A	109.00	C11—C20—H20A	109.00
C1—C10—H10B	109.00	C11—C20—H20B	109.00
C9—C10—H10A	109.00	C19—C20—H20A	109.00
C9—C10—H10B	109.00	C19—C20—H20B	109.00
H10A—C10—H10B	108.00	H20A—C20—H20B	108.00

C6—C1—C2—C3	−0.3 (4)	C16—C11—C12—C13	1.7 (4)
C10—C1—C2—C3	177.9 (3)	C20—C11—C12—C13	−176.3 (3)
C2—C1—C6—C5	−0.6 (3)	C12—C11—C16—C15	−1.8 (3)
C2—C1—C6—C7	−178.9 (2)	C12—C11—C16—C17	177.3 (2)
C10—C1—C6—C5	−178.8 (2)	C20—C11—C16—C15	176.2 (2)
C10—C1—C6—C7	2.9 (3)	C20—C11—C16—C17	−4.7 (3)
C2—C1—C10—C9	156.0 (2)	C12—C11—C20—C19	−157.1 (2)
C6—C1—C10—C9	−25.8 (3)	C16—C11—C20—C19	25.0 (3)
C1—C2—C3—C4	0.5 (5)	C11—C12—C13—C14	−0.3 (5)
C2—C3—C4—C5	0.2 (5)	C12—C13—C14—C15	−1.0 (5)
C3—C4—C5—C6	−1.1 (4)	C13—C14—C15—C16	0.9 (5)
C4—C5—C6—C1	1.3 (4)	C14—C15—C16—C11	0.6 (4)
C4—C5—C6—C7	179.6 (2)	C14—C15—C16—C17	−178.6 (3)
C1—C6—C7—O1	174.4 (2)	C11—C16—C17—O2	−171.0 (2)
C1—C6—C7—C8	−4.7 (3)	C11—C16—C17—C18	8.1 (3)
C5—C6—C7—O1	−4.0 (3)	C15—C16—C17—O2	8.1 (3)
C5—C6—C7—C8	177.1 (2)	C15—C16—C17—C18	−172.9 (2)
O1—C7—C8—I1A	87.3 (2)	O2—C17—C18—I2A	−89.8 (2)
O1—C7—C8—C9	−149.8 (2)	O2—C17—C18—C19	147.6 (2)
C6—C7—C8—I1A	−93.7 (2)	C16—C17—C18—I2A	91.2 (2)
C6—C7—C8—C9	29.3 (3)	C16—C17—C18—C19	−31.5 (3)
I1A—C8—C9—C10	66.6 (3)	I2A—C18—C19—C20	−65.9 (3)
C7—C8—C9—C10	−52.2 (3)	C17—C18—C19—C20	52.1 (3)
C8—C9—C10—C1	50.1 (3)	C18—C19—C20—C11	−48.6 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···*Cg*3	0.93	2.95	3.700 (4)	139