N-formyl-N '-(2-oxidobenzylidene) hydrazine-kappa(3) O, N, O '] diphenyltin(IV)

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[*N*-Formyl-*N*′-(2-oxidobenzylidene)hydrazine-κ^3O,N,O']diphenyltin(IV)

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The title compound, $[Sn(C_6H_5)_2(C_8H_6N_2O_2)]$, features a five-coordinate C_2NO_2 coordination geometry for Sn that is intermediate between trigonal-bipyramidal and square-pyramidal.

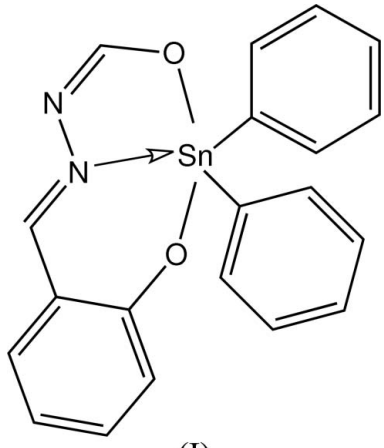
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
R factor = 0.022
wR factor = 0.058
Data-to-parameter ratio = 14.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The title compound, (I), was investigated as a part of an ongoing investigation of the putative biological activities of organotin compounds (Rehman *et al.*, 2004; Gielen & Tiekink, 2005).



(I)

In (I) (Fig. 1 and Table 1), the Sn atom is coordinated by two O atoms and an N atom from the tridentate ligand and two *ipso*-C atoms belonging to the two phenyl substituents. The tin coordination is highly distorted, as seen in the value of τ = 0.56, indicating a geometry biased towards trigonal bipyramidal (τ = 1.0) rather than square pyramidal (τ = 0.0; Addison *et al.*, 1984). The distortion can be rationalized in terms of the steric demands of the two chelate rings formed by the tridentate ligand. Similar structures have been found in the analogues in which the C1 position in (I) is substituted with Me (τ = 0.59; Diouf *et al.*, 1999) and Ph (τ = 0.50 and 0.55 for the two independent molecules; Dey *et al.*, 2003). The dihedral angles between the mean plane of the ligand and the C9 and C15 phenyl rings are 68.82 (9)° and 56.13 (10)°, respectively. The angle between the C9 and C15 ring planes is 58.26 (12)°.

In the crystal structure of (I), centrosymmetric pairs of molecules associate via C—H···π interactions (Table 2). Such an arrangement effectively blocks off both N atoms as well as the O2 atom from forming intermolecular contacts. The loosely associated dimers are connected into chains via C—H···O contacts (Fig. 2 and Table 2).

Experimental

N-(2-Hydroxybenzylidene)formylhydrazide (2.5 mmol, 0.41 g) and Et_3N (5 mmol, 0.7 ml) were added to dry toluene (100 ml) in a round-bottom flask equipped with a reflux condenser. Diphenyltin(IV) dichloride (2.5 mmol, 0.86 g) dissolved in dry toluene (20 ml) was then added. The reaction mixture was stirred at room temperature for 5 h and allowed to stand overnight. The Et_3N·HCl that formed was filtered off and the clear yellow solution was evaporated on a rotary evaporator under reduced pressure. Crystals of (I) suitable for single crystal analysis were obtained by recrystallization from a chloroform solution; m.p = 389–391 K.

Crystal data

$[Sn(C_6H_5)_2(C_8H_6N_2O_2)]$
M_r = 435.04
Triclinic, $P\overline{1}$
a = 8.9903 (5) Å
b = 9.0639 (5) Å
c = 11.8622 (7) Å
α = 77.277 (1)°
β = 74.324 (1)°
γ = 68.869 (1)°
V = 859.93 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.50 mm^{-1}
T = 100 (2) K
0.49 × 0.43 × 0.39 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2006)
T_{min} = 0.459, T_{max} = 0.557
6594 measured reflections
3297 independent reflections
3196 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.058
S = 1.12
3297 reflections
226 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.68 e Å^{-3}
$\Delta\rho_{min}$ = −0.74 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn—O1	2.1470 (18)	Sn—C15	2.125 (2)
Sn—O2	2.0654 (17)	N1—C1	1.299 (3)
Sn—N2	2.162 (2)	N1—N2	1.411 (3)
Sn—C9	2.120 (2)	N2—C2	1.299 (3)
O1—Sn—O2	158.42 (7)	O2—Sn—N2	84.49 (7)
O1—Sn—N2	73.94 (7)	C9—Sn—C15	124.62 (9)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···Cg^{i}	0.95	2.93	3.872 (3)	172
C19—H19···O1^{ii}	0.95	2.67	3.554 (3)	154

Symmetry codes: (i) $-x, -y, -z+1$; (ii) $-x+1, -y, -z$. *Cg* is the centroid of the C9–C14 ring.

H atoms were positioned geometrically (C—H = 0.95 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 2006); cell refinement: *SAINT-Plus* (Bruker, 2006); data reduction: *SAINT-Plus*; program(s) used to solve structure: *PATTY* in *DIRDIF92* (Beurskens *et al.*, 1992); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997);

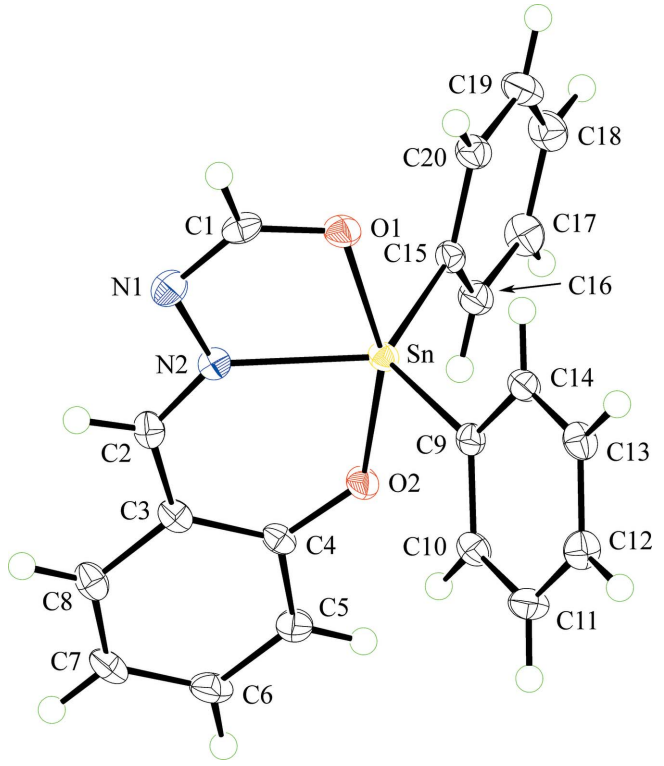


Figure 1
The molecular structure of (I), showing the atom labelling and displacement ellipsoids drawn at the 50% probability level (arbitrary spheres for the H atoms).

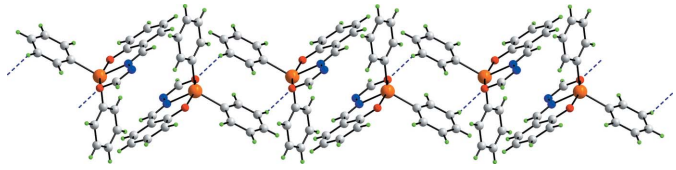


Figure 2
Chain formation via C—H···O (blue dashed lines) and C—H···π interactions in the crystal structure of (I). Colour code: Sn atoms are shown in orange, O atoms in red, N atom in blue, C atoms in grey and H atoms in green.

molecular graphics: *ORTEPII* (Johnson, 1976) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXL97*.

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(I)

Crystal data

$C_{20}H_{16}N_2O_2Sn$
$M_r = 435.04$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.9903$ (5) Å
$b = 9.0639$ (5) Å
$c = 11.8622$ (7) Å
$\alpha = 77.277$ (1)°
$\beta = 74.324$ (1)°
$\gamma = 68.869$ (1)°
$V = 859.93$ (8) Å^3
$Z = 2$
$F(000) = 432$
$D_x = 1.680$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7180 reflections
$\theta = 2.4$–29.6°
$\mu = 1.50$ mm^{-1}
$T = 100$ K
Block, yellow
0.49 × 0.43 × 0.39 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 4096x4096 / 62x62 (binned 512) pixels mm^{-1}
ω scans
Absorption correction: multi-scan SADABS (Bruker, 2006)
$T_{min} = 0.459$, $T_{max} = 0.557$
6594 measured reflections
3297 independent reflections
3196 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.4°$
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 10$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.058$
$S = 1.12$
3297 reflections
226 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
$w = 1/[\sigma^2(F_o^2) + (0.0258P)^2 + 0.9175P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.68$ e Å^{-3}
$\Delta\rho_{min} = -0.74$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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}
Sn	0.073101 (18)	0.179092 (18)	0.246058 (13)	0.01552 (7)
O1	0.2676 (2)	−0.0444 (2)	0.24888 (15)	0.0215 (4)
O2	−0.1012 (2)	0.3823 (2)	0.30705 (15)	0.0201 (4)
N1	0.2437 (3)	−0.0236 (3)	0.44564 (19)	0.0217 (4)
N2	0.1199 (2)	0.1213 (2)	0.42263 (17)	0.0166 (4)
C1	0.3058 (3)	−0.0949 (3)	0.3519 (2)	0.0195 (5)
H1	0.3885	−0.1954	0.3595	0.023*
C2	0.0467 (3)	0.2021 (3)	0.5111 (2)	0.0182 (5)
H2	0.0854	0.1596	0.5823	0.022*
C3	−0.0863 (3)	0.3487 (3)	0.5121 (2)	0.0176 (5)
C4	−0.1546 (3)	0.4316 (3)	0.4123 (2)	0.0169 (5)
C5	−0.2846 (3)	0.5750 (3)	0.4244 (2)	0.0219 (5)
H5	−0.3305	0.6324	0.3579	0.026*
C6	−0.3469 (3)	0.6339 (3)	0.5313 (2)	0.0251 (6)
H6	−0.4350	0.7310	0.5376	0.030*
C7	−0.2814 (3)	0.5518 (3)	0.6308 (2)	0.0228 (5)
H7	−0.3255	0.5921	0.7044	0.027*
C8	−0.1529 (3)	0.4126 (3)	0.6205 (2)	0.0209 (5)
H8	−0.1075	0.3578	0.6877	0.025*
C9	−0.0923 (3)	0.0795 (3)	0.2184 (2)	0.0169 (5)
C10	−0.2589 (3)	0.1418 (3)	0.2658 (2)	0.0198 (5)
H10	−0.2980	0.2304	0.3083	0.024*
C11	−0.3678 (3)	0.0742 (3)	0.2509 (2)	0.0239 (5)
H11	−0.4807	0.1154	0.2847	0.029*
C12	−0.3118 (3)	−0.0530 (3)	0.1867 (2)	0.0232 (5)
H12	−0.3867	−0.0980	0.1759	0.028*
C13	−0.1469 (3)	−0.1150 (3)	0.1383 (2)	0.0208 (5)
H13	−0.1090	−0.2018	0.0940	0.025*
C14	−0.0366 (3)	−0.0496 (3)	0.1545 (2)	0.0190 (5)
H14	0.0764	−0.0928	0.1222	0.023*
C15	0.2105 (3)	0.3034 (3)	0.1105 (2)	0.0167 (5)
C16	0.1485 (3)	0.4688 (3)	0.0857 (2)	0.0219 (5)
H16	0.0481	0.5254	0.1320	0.026*
C17	0.2329 (3)	0.5521 (3)	−0.0067 (2)	0.0259 (6)
H17	0.1902	0.6650	−0.0233	0.031*
C18	0.3795 (3)	0.4691 (3)	−0.0743 (2)	0.0265 (6)
H18	0.4366	0.5255	−0.1378	0.032*
C19	0.4429 (3)	0.3049 (3)	−0.0498 (2)	0.0269 (6)
H19	0.5438	0.2490	−0.0960	0.032*
C20	0.3588 (3)	0.2211 (3)	0.0427 (2)	0.0214 (5)

H20	0.4025	0.1083	0.0595	0.026*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn	0.01591 (10)	0.01400 (10)	0.01511 (9)	−0.00329 (7)	−0.00194 (6)	−0.00308 (6)
O1	0.0194 (8)	0.0179 (9)	0.0227 (9)	−0.0005 (7)	−0.0027 (7)	−0.0052 (7)
O2	0.0207 (8)	0.0184 (9)	0.0173 (8)	−0.0005 (7)	−0.0038 (7)	−0.0050 (7)
N1	0.0217 (11)	0.0159 (11)	0.0248 (11)	−0.0023 (9)	−0.0081 (9)	0.0001 (9)
N2	0.0176 (10)	0.0138 (10)	0.0182 (10)	−0.0059 (8)	−0.0041 (8)	−0.0003 (8)
C1	0.0153 (11)	0.0123 (12)	0.0278 (13)	−0.0025 (10)	−0.0057 (10)	0.0015 (10)
C2	0.0236 (12)	0.0185 (12)	0.0157 (11)	−0.0127 (10)	−0.0039 (9)	0.0011 (9)
C3	0.0206 (12)	0.0166 (12)	0.0172 (11)	−0.0102 (10)	0.0003 (9)	−0.0034 (9)
C4	0.0181 (11)	0.0165 (12)	0.0161 (11)	−0.0079 (10)	0.0009 (9)	−0.0041 (9)
C5	0.0195 (12)	0.0207 (13)	0.0232 (12)	−0.0027 (10)	−0.0039 (10)	−0.0054 (10)
C6	0.0183 (12)	0.0259 (14)	0.0295 (14)	−0.0045 (11)	0.0020 (10)	−0.0134 (11)
C7	0.0251 (13)	0.0261 (14)	0.0182 (12)	−0.0125 (11)	0.0050 (10)	−0.0098 (10)
C8	0.0271 (13)	0.0211 (13)	0.0163 (11)	−0.0131 (11)	−0.0010 (10)	−0.0017 (10)
C9	0.0185 (11)	0.0166 (12)	0.0137 (10)	−0.0049 (10)	−0.0037 (9)	0.0009 (9)
C10	0.0218 (12)	0.0185 (12)	0.0179 (11)	−0.0053 (10)	−0.0016 (9)	−0.0054 (10)
C11	0.0182 (12)	0.0267 (14)	0.0256 (13)	−0.0076 (11)	0.0008 (10)	−0.0074 (11)
C12	0.0257 (13)	0.0227 (14)	0.0243 (13)	−0.0110 (11)	−0.0060 (10)	−0.0030 (11)
C13	0.0267 (13)	0.0153 (12)	0.0199 (12)	−0.0048 (10)	−0.0044 (10)	−0.0057 (10)
C14	0.0193 (12)	0.0185 (12)	0.0158 (11)	−0.0033 (10)	−0.0019 (9)	−0.0025 (9)
C15	0.0184 (11)	0.0184 (12)	0.0149 (11)	−0.0071 (10)	−0.0031 (9)	−0.0036 (9)
C16	0.0219 (12)	0.0214 (13)	0.0192 (12)	−0.0038 (11)	−0.0030 (10)	−0.0031 (10)
C17	0.0314 (14)	0.0203 (14)	0.0249 (13)	−0.0102 (12)	−0.0057 (11)	0.0019 (11)
C18	0.0296 (14)	0.0297 (15)	0.0221 (13)	−0.0164 (12)	−0.0016 (11)	−0.0007 (11)
C19	0.0228 (13)	0.0328 (16)	0.0246 (13)	−0.0110 (12)	0.0037 (11)	−0.0104 (12)
C20	0.0211 (12)	0.0214 (13)	0.0218 (12)	−0.0060 (11)	−0.0028 (10)	−0.0070 (10)

Geometric parameters (Å, º)

Sn—O1	2.1470 (18)	C9—C10	1.397 (3)
Sn—O2	2.0654 (17)	C9—C14	1.399 (3)
Sn—N2	2.162 (2)	C10—C11	1.392 (4)
Sn—C9	2.120 (2)	C10—H10	0.9500
Sn—C15	2.125 (2)	C11—C12	1.387 (4)
O1—C1	1.299 (3)	C11—H11	0.9500
O2—C4	1.325 (3)	C12—C13	1.388 (4)
N1—C1	1.299 (3)	C12—H12	0.9500
N1—N2	1.411 (3)	C13—C14	1.396 (4)
N2—C2	1.299 (3)	C13—H13	0.9500
C1—H1	0.9500	C14—H14	0.9500
C2—C3	1.431 (4)	C15—C16	1.391 (4)
C2—H2	0.9500	C15—C20	1.396 (3)
C3—C4	1.418 (3)	C16—C17	1.395 (4)
C3—C8	1.418 (3)	C16—H16	0.9500

C4—C5	1.405 (3)	C17—C18	1.387 (4)
C5—C6	1.381 (4)	C17—H17	0.9500
C5—H5	0.9500	C18—C19	1.383 (4)
C6—C7	1.402 (4)	C18—H18	0.9500
C6—H6	0.9500	C19—C20	1.397 (4)
C7—C8	1.371 (4)	C19—H19	0.9500
C7—H7	0.9500	C20—H20	0.9500
C8—H8	0.9500		

O2—Sn—C9	96.23 (8)	C7—C8—H8	119.2
O2—Sn—C15	95.10 (8)	C3—C8—H8	119.2
O1—Sn—O2	158.42 (7)	C10—C9—C14	119.4 (2)
O1—Sn—N2	73.94 (7)	C10—C9—Sn	119.82 (18)
O2—Sn—N2	84.49 (7)	C14—C9—Sn	120.82 (17)
C9—Sn—C15	124.62 (9)	C11—C10—C9	120.2 (2)
C9—Sn—O1	93.72 (8)	C11—C10—H10	119.9
C15—Sn—O1	94.91 (8)	C9—C10—H10	119.9
C9—Sn—N2	115.22 (8)	C12—C11—C10	120.1 (2)
C15—Sn—N2	119.75 (8)	C12—C11—H11	119.9
C1—O1—Sn	112.38 (15)	C10—C11—H11	119.9
C4—O2—Sn	132.88 (16)	C13—C12—C11	120.3 (2)
C1—N1—N2	110.2 (2)	C13—C12—H12	119.9
C2—N2—N1	115.7 (2)	C11—C12—H12	119.9
C2—N2—Sn	128.20 (17)	C12—C13—C14	119.9 (2)
N1—N2—Sn	116.09 (14)	C12—C13—H13	120.0
O1—C1—N1	127.4 (2)	C14—C13—H13	120.0
O1—C1—H1	116.3	C13—C14—C9	120.1 (2)
N1—C1—H1	116.3	C13—C14—H14	119.9
N2—C2—C3	126.6 (2)	C9—C14—H14	119.9
N2—C2—H2	116.7	C16—C15—C20	119.5 (2)
C3—C2—H2	116.7	C16—C15—Sn	119.48 (17)
C4—C3—C8	118.8 (2)	C20—C15—Sn	120.95 (18)
C4—C3—C2	124.0 (2)	C15—C16—C17	120.4 (2)
C8—C3—C2	117.1 (2)	C15—C16—H16	119.8
O2—C4—C5	117.7 (2)	C17—C16—H16	119.8
O2—C4—C3	123.6 (2)	C18—C17—C16	119.7 (3)
C5—C4—C3	118.7 (2)	C18—C17—H17	120.2
C6—C5—C4	121.0 (2)	C16—C17—H17	120.2
C6—C5—H5	119.5	C19—C18—C17	120.4 (2)
C4—C5—H5	119.5	C19—C18—H18	119.8
C5—C6—C7	120.7 (2)	C17—C18—H18	119.8
C5—C6—H6	119.7	C18—C19—C20	120.1 (2)
C7—C6—H6	119.7	C18—C19—H19	119.9
C8—C7—C6	119.2 (2)	C20—C19—H19	119.9
C8—C7—H7	120.4	C15—C20—C19	119.9 (2)
C6—C7—H7	120.4	C15—C20—H20	120.1
C7—C8—C3	121.5 (2)	C19—C20—H20	120.1

O2—Sn—O1—C1	3.5 (3)	C4—C3—C8—C7	0.2 (4)
C9—Sn—O1—C1	−113.83 (16)	C2—C3—C8—C7	−179.5 (2)
C15—Sn—O1—C1	120.93 (16)	O2—Sn—C9—C10	−13.3 (2)
N2—Sn—O1—C1	1.36 (15)	C15—Sn—C9—C10	−113.89 (19)
C9—Sn—O2—C4	110.4 (2)	O1—Sn—C9—C10	147.53 (19)
C15—Sn—O2—C4	−123.9 (2)	N2—Sn—C9—C10	73.5 (2)
O1—Sn—O2—C4	−6.5 (3)	O2—Sn—C9—C14	167.59 (19)
N2—Sn—O2—C4	−4.4 (2)	C15—Sn—C9—C14	67.0 (2)
C1—N1—N2—C2	178.9 (2)	O1—Sn—C9—C14	−31.6 (2)
C1—N1—N2—Sn	−0.2 (2)	N2—Sn—C9—C14	−105.59 (19)
O2—Sn—N2—C2	1.3 (2)	C14—C9—C10—C11	0.9 (4)
C9—Sn—N2—C2	−93.0 (2)	Sn—C9—C10—C11	−178.21 (19)
C15—Sn—N2—C2	94.0 (2)	C9—C10—C11—C12	−1.4 (4)
O1—Sn—N2—C2	−179.5 (2)	C10—C11—C12—C13	0.8 (4)
O2—Sn—N2—N1	−179.85 (16)	C11—C12—C13—C14	0.3 (4)
C9—Sn—N2—N1	85.85 (17)	C12—C13—C14—C9	−0.8 (4)
C15—Sn—N2—N1	−87.10 (17)	C10—C9—C14—C13	0.1 (4)
O1—Sn—N2—N1	−0.65 (14)	Sn—C9—C14—C13	179.27 (18)
Sn—O1—C1—N1	−2.3 (3)	O2—Sn—C15—C16	−8.3 (2)
N2—N1—C1—O1	1.7 (3)	C9—Sn—C15—C16	92.9 (2)
N1—N2—C2—C3	−177.4 (2)	O1—Sn—C15—C16	−169.15 (19)
Sn—N2—C2—C3	1.5 (3)	N2—Sn—C15—C16	−94.9 (2)
N2—C2—C3—C4	−2.6 (4)	O2—Sn—C15—C20	174.43 (19)
N2—C2—C3—C8	177.1 (2)	C9—Sn—C15—C20	−84.4 (2)
Sn—O2—C4—C5	−176.08 (16)	O1—Sn—C15—C20	13.6 (2)
Sn—O2—C4—C3	4.7 (3)	N2—Sn—C15—C20	87.9 (2)
C8—C3—C4—O2	179.9 (2)	C20—C15—C16—C17	0.5 (4)
C2—C3—C4—O2	−0.5 (4)	Sn—C15—C16—C17	−176.82 (19)
C8—C3—C4—C5	0.7 (3)	C15—C16—C17—C18	0.1 (4)
C2—C3—C4—C5	−179.7 (2)	C16—C17—C18—C19	−0.6 (4)
O2—C4—C5—C6	179.9 (2)	C17—C18—C19—C20	0.6 (4)
C3—C4—C5—C6	−0.8 (4)	C16—C15—C20—C19	−0.5 (4)
C4—C5—C6—C7	0.1 (4)	Sn—C15—C20—C19	176.75 (19)
C5—C6—C7—C8	0.8 (4)	C18—C19—C20—C15	0.0 (4)
C6—C7—C8—C3	−0.9 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···*Cg*^{i}	0.95	2.93	3.872 (3)	172
C19—H19···O1^{ii}	0.95	2.67	3.554 (3)	154

Symmetry codes: (i) −*x*, −*y*, −*z*+1; (ii) −*x*+1, −*y*, −*z*.