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# {4-Chloro-*N*′-[(2-oxidonaphthalen-1-yl-κ*O*)methylidene]benzohydrazidato-κ$^2$*N*′,*O*}dimethyltin(IV)

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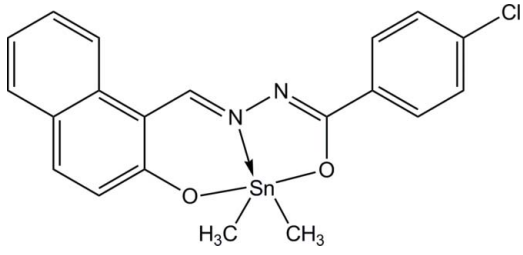
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.007 Å; $R$ factor = 0.040; $wR$ factor = 0.107; data-to-parameter ratio = 14.1.

In the title complex, $[Sn(CH_3)_2(C_{18}H_{11}ClN_2O_2)]$, the $Sn^{IV}$ ion is coordinated by two O atoms and an N atom from a 4-chloro-*N*′-[(2-oxidonaphthalen-1-yl)methylidene]benzohydrazidate ligand and two C atoms from two methyl ligands in a distorted trigonal–bipyramidal geometry [Sn—O = 2.092 (3) and 2.144 (3) Å; Sn—N = 2.160 (4) Å]. The dihedral angle between the naphthalene ring system and the benzene ring is 8.6 (2)°. In the crystal, adjacent molecules are linked by weak C—H···O hydrogen bonds, forming a chain along the *b*-axis direction.

## Related literature

For the biological activity and related structures of organotin compounds, see: Hong *et al.* (2013).




## Experimental

### Crystal data

$[Sn(CH_3)_2(C_{18}H_{11}ClN_2O_2)]$
$M_r$ = 471.50
Monoclinic, $P2_1/n$
$a$ = 8.7927 (8) Å
$b$ = 17.4170 (15) Å
$c$ = 12.5014 (12) Å
$\beta$ = 98.690 (9)°
$V$ = 1892.5 (3) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 1.51 mm$^{-1}$
$T$ = 293 K
0.24 × 0.23 × 0.13 mm

### Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.714, $T_{max}$ = 0.828
11127 measured reflections
3343 independent reflections
2584 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.047

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.107
$S$ = 1.06
3343 reflections
237 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.83 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.61 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C5—H5···O2$^{i}$ | 0.93 | 2.54 | 3.388 (6) | 152 |

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5669).

## References

Hong, M., Yin, H., Zhang, X., Li, C., Yue, C. & Cheng, S. (2013). *J. Organomet. Chem.*, 724, 23–31.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

# supporting information

*Acta Cryst.* (2013). E**69**, m688 [doi:10.1107/S1600536813031826]

## {4-Chloro-*N*′-[(2-oxidonaphthalen-1-yl-$\kappa O$)methylidene]benzohydrazidato-$\kappa^2 N',O$}dimethyltin(IV)

**Jichun Cui, Yanling Qiao and Fei Wang**

### S1. Comment

The chemistry of organotin(IV) derivatives is a subject of study with growing interest due to their significant antimicrobial properties as well as antitumor activities (Hong *et al.*, 2013). As a part of our ongoing investigations in this field we have synthesized the title compound and present its crystal structure here. The molecular structure of the title compound, (I), is shown in Fig. 1. The Sn atom has distorted trigonal-bipyramidal geometry, with atoms O1 and O2 in axial positions [O1—Sn1—O2 = 153.39 (13) °] and the atoms C19, C20 and N2 in equatorial positions. The sum of the equatorial angles is 359.9 °, indicating approximate coplanarity for these atoms. The Sn1—N2 bond length is 2.160 (4) Å close to the sum of the non-polar covalent radii 2.15 Å, indicating a strong Sn—N interaction. The O atoms coordinate to the Sn atom with one shorter [2.092 (3) Å] and one longer [2.144 (3) Å] bond. The dihedral angle between the naphthalene ring system (C9-C18) and the benzene ring (C1-C6) is 8.6 (2)°.

### S2. Experimental

2-hydroxy-1-naphthaldehyde 4-chlorobenzoylhydrazone (1 mmol) and sodium ethoxide (1 mmol) were added to the solution of dry methanol(30 ml) and stirred for 10 mins. Dimeyltin dichloride (1 mmol) was then added to the reactor and the reaction mixture was stirred for 4 h. The resulting clear solution was evaporated under vacuum. The product was crystallized from a mixture of dichloromethane/ethanol(1:1) to yield orange blocks of the title compound (yield 73%).

### S3. Refinement

The H atoms were positioned geometrically, with methyl C—H distances of 0.96 Å and aromatic and aldehydic C—H distances of 0.93 Å, and refined as riding on their parent atoms, with $U_{iso}$(H) = 1.2 $U_{eq}$(C) or 1.5 $U_{eq}$(C) for the methyl groups.

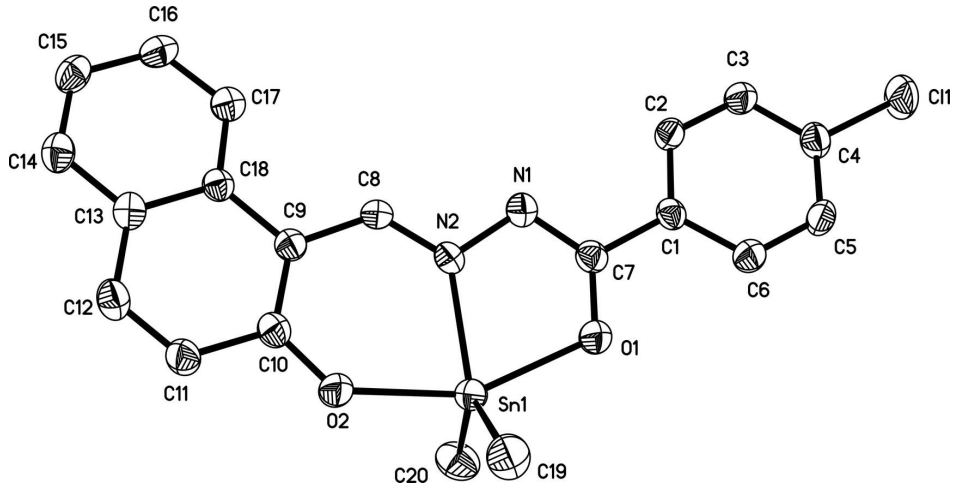


**Figure 1**

The molecular structure of the title compound, showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

**{4-Chloro-*N*′-[(2-oxidonaphthalen-1-yl-$\kappa O$)methylidene]benzohydrazidato-$\kappa^2 N', O$}dimethyltin(IV)**

*Crystal data*

$[Sn(CH_3)_2(C_{18}H_{11}ClN_2O_2)]$
$M_r$ = 471.50
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a$ = 8.7927 (8) Å
$b$ = 17.4170 (15) Å
$c$ = 12.5014 (12) Å
$\beta$ = 98.690 (9)°
$V$ = 1892.5 (3) Å$^3$
$Z$ = 4
$F(000)$ = 936
$D_x$ = 1.655 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 2600 reflections
$\theta$ = 2.6–28.5°
$\mu$ = 1.51 mm$^{-1}$
$T$ = 293 K
Block, orange
0.24 × 0.23 × 0.13 mm

*Data collection*

Siemens SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.714, $T_{max}$ = 0.828
11127 measured reflections
3343 independent reflections
2584 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.047
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 2.6°
$h$ = −8→10
$k$ = −20→19
$l$ = −14→14

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.107
$S$ = 1.06
3343 reflections
237 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
$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 0.4409P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.83 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.61 e Å$^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Sn1 | −0.14514 (4) | 0.026153 (19) | 0.73069 (3) | 0.04369 (15) |
| O1 | −0.1839 (4) | 0.1313 (2) | 0.8117 (3) | 0.0542 (9) |
| O2 | −0.0073 (4) | −0.0641 (2) | 0.6906 (3) | 0.0597 (10) |
| N1 | 0.0353 (5) | 0.1015 (2) | 0.9316 (3) | 0.0511 (10) |
| N2 | 0.0421 (5) | 0.0380 (2) | 0.8630 (3) | 0.0447 (10) |
| C1 | −0.1055 (5) | 0.2148 (3) | 0.9621 (4) | 0.0408 (11) |
| C2 | −0.0132 (5) | 0.2290 (3) | 1.0615 (4) | 0.0457 (12) |
| H2 | 0.0645 | 0.1945 | 1.0874 | 0.055* |
| C3 | −0.0355 (6) | 0.2931 (3) | 1.1216 (4) | 0.0532 (13) |
| H3 | 0.0261 | 0.3019 | 1.1878 | 0.064* |
| C4 | −0.1512 (6) | 0.3443 (3) | 1.0820 (4) | 0.0528 (13) |
| C5 | −0.2422 (6) | 0.3321 (3) | 0.9852 (4) | 0.0534 (13) |
| H5 | −0.3192 | 0.3671 | 0.9599 | 0.064* |
| C6 | −0.2204 (6) | 0.2679 (3) | 0.9248 (4) | 0.0503 (12) |
| H6 | −0.2826 | 0.2599 | 0.8587 | 0.060* |
| C7 | −0.0849 (5) | 0.1448 (3) | 0.8972 (4) | 0.0446 (11) |
| C8 | 0.1604 (5) | −0.0072 (3) | 0.8915 (4) | 0.0420 (11) |
| H8 | 0.2257 | 0.0062 | 0.9543 | 0.050* |
| C9 | 0.2008 (5) | −0.0748 (3) | 0.8370 (4) | 0.0411 (11) |
| C10 | 0.1179 (6) | −0.0978 (3) | 0.7378 (4) | 0.0466 (12) |
| C11 | 0.1701 (6) | −0.1620 (3) | 0.6826 (4) | 0.0530 (13) |
| H11 | 0.1140 | −0.1776 | 0.6170 | 0.064* |
| C12 | 0.2989 (6) | −0.2008 (3) | 0.7233 (4) | 0.0555 (13) |
| H12 | 0.3304 | −0.2420 | 0.6847 | 0.067* |
| C13 | 0.3880 (5) | −0.1797 (3) | 0.8248 (4) | 0.0454 (12) |
| C14 | 0.5222 (6) | −0.2202 (3) | 0.8669 (4) | 0.0543 (13) |
| H14 | 0.5554 | −0.2602 | 0.8268 | 0.065* |
| C15 | 0.6041 (6) | −0.2024 (3) | 0.9642 (4) | 0.0551 (13) |
| H15 | 0.6932 | −0.2294 | 0.9904 | 0.066* |
| C16 | 0.5533 (6) | −0.1430 (3) | 1.0250 (4) | 0.0539 (13) |
| H16 | 0.6072 | −0.1315 | 1.0930 | 0.065* |
| C17 | 0.4242 (5) | −0.1012 (3) | 0.9852 (4) | 0.0478 (12) |
| H17 | 0.3932 | −0.0614 | 1.0267 | 0.057* |
| C18 | 0.3378 (5) | −0.1170 (3) | 0.8833 (4) | 0.0413 (11) |
| C19 | −0.1543 (7) | 0.0779 (4) | 0.5774 (4) | 0.0695 (16) |
| H19A | −0.0524 | 0.0920 | 0.5662 | 0.104* |
| H19B | −0.2178 | 0.1229 | 0.5739 | 0.104* |
| H19C | −0.1970 | 0.0423 | 0.5223 | 0.104* |
| C20 | −0.3234 (7) | −0.0415 (4) | 0.7770 (5) | 0.0732 (18) |
| H20A | −0.3626 | −0.0755 | 0.7189 | 0.110* |
| H20B | −0.4047 | −0.0088 | 0.7932 | 0.110* |
| H20C | −0.2841 | −0.0711 | 0.8399 | 0.110* |
| Cl1 | −0.1839 (2) | 0.42381 (10) | 1.16029 (14) | 0.0880 (5) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Sn1 | 0.0436 (2) | 0.0429 (2) | 0.0436 (2) | −0.00084 (15) | 0.00346 (16) | −0.00068 (16) |
| O1 | 0.0518 (19) | 0.054 (2) | 0.054 (2) | 0.0098 (17) | 0.0001 (17) | −0.0102 (18) |
| O2 | 0.057 (2) | 0.067 (2) | 0.050 (2) | 0.017 (2) | −0.0076 (17) | −0.0145 (19) |
| N1 | 0.048 (2) | 0.047 (3) | 0.059 (3) | 0.004 (2) | 0.006 (2) | −0.007 (2) |
| N2 | 0.048 (2) | 0.039 (2) | 0.046 (2) | 0.0002 (18) | 0.0058 (19) | −0.0097 (19) |
| C1 | 0.042 (2) | 0.034 (3) | 0.048 (3) | −0.004 (2) | 0.010 (2) | 0.000 (2) |
| C2 | 0.040 (3) | 0.044 (3) | 0.053 (3) | 0.005 (2) | 0.006 (2) | 0.002 (2) |
| C3 | 0.051 (3) | 0.056 (3) | 0.050 (3) | 0.006 (3) | −0.001 (2) | −0.006 (3) |
| C4 | 0.060 (3) | 0.044 (3) | 0.055 (3) | 0.007 (3) | 0.010 (3) | −0.005 (3) |
| C5 | 0.057 (3) | 0.049 (3) | 0.051 (3) | 0.018 (3) | 0.000 (3) | 0.001 (3) |
| C6 | 0.052 (3) | 0.050 (3) | 0.046 (3) | 0.005 (2) | 0.000 (2) | 0.001 (3) |
| C7 | 0.045 (3) | 0.040 (3) | 0.048 (3) | −0.004 (2) | 0.008 (2) | 0.000 (2) |
| C8 | 0.040 (3) | 0.044 (3) | 0.040 (3) | −0.001 (2) | 0.001 (2) | −0.003 (2) |
| C9 | 0.040 (2) | 0.041 (3) | 0.043 (3) | −0.002 (2) | 0.009 (2) | −0.001 (2) |
| C10 | 0.050 (3) | 0.046 (3) | 0.045 (3) | 0.000 (2) | 0.011 (2) | −0.004 (2) |
| C11 | 0.064 (3) | 0.052 (3) | 0.041 (3) | 0.009 (3) | 0.002 (2) | −0.005 (3) |
| C12 | 0.066 (3) | 0.050 (3) | 0.052 (3) | 0.008 (3) | 0.015 (3) | −0.006 (3) |
| C13 | 0.049 (3) | 0.043 (3) | 0.047 (3) | 0.002 (2) | 0.015 (2) | 0.005 (2) |
| C14 | 0.058 (3) | 0.045 (3) | 0.062 (3) | 0.007 (3) | 0.013 (3) | 0.004 (3) |
| C15 | 0.048 (3) | 0.052 (3) | 0.066 (3) | 0.007 (2) | 0.007 (3) | 0.014 (3) |
| C16 | 0.044 (3) | 0.060 (3) | 0.055 (3) | −0.003 (3) | −0.002 (2) | 0.009 (3) |
| C17 | 0.050 (3) | 0.046 (3) | 0.048 (3) | −0.003 (2) | 0.009 (2) | 0.005 (2) |
| C18 | 0.044 (3) | 0.037 (3) | 0.044 (3) | −0.003 (2) | 0.008 (2) | 0.002 (2) |
| C19 | 0.091 (4) | 0.066 (4) | 0.051 (3) | 0.007 (3) | 0.008 (3) | 0.008 (3) |
| C20 | 0.067 (4) | 0.073 (4) | 0.082 (5) | −0.025 (3) | 0.018 (4) | −0.008 (3) |
| Cl1 | 0.1110 (13) | 0.0687 (10) | 0.0780 (10) | 0.0321 (9) | −0.0065 (9) | −0.0259 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Sn1—O2 | 2.092 (3) | C9—C10 | 1.399 (7) |
| Sn1—C19 | 2.108 (5) | C9—C18 | 1.455 (6) |
| Sn1—C20 | 2.111 (5) | C10—C11 | 1.426 (7) |
| Sn1—O1 | 2.144 (3) | C11—C12 | 1.351 (7) |
| Sn1—N2 | 2.160 (4) | C11—H11 | 0.9300 |
| O1—C7 | 1.294 (5) | C12—C13 | 1.435 (7) |
| O2—C10 | 1.306 (6) | C12—H12 | 0.9300 |
| N1—C7 | 1.317 (6) | C13—C14 | 1.406 (7) |
| N1—N2 | 1.407 (5) | C13—C18 | 1.422 (6) |
| N2—C8 | 1.310 (6) | C14—C15 | 1.353 (7) |
| C1—C6 | 1.397 (7) | C14—H14 | 0.9300 |
| C1—C2 | 1.400 (7) | C15—C16 | 1.397 (7) |
| C1—C7 | 1.489 (6) | C15—H15 | 0.9300 |
| C2—C3 | 1.377 (7) | C16—C17 | 1.377 (7) |
| C2—H2 | 0.9300 | C16—H16 | 0.9300 |
| C3—C4 | 1.387 (7) | C17—C18 | 1.408 (6) |

| | | | |
|---|---|---|---|
| C3—H3 | 0.9300 | C17—H17 | 0.9300 |
| C4—C5 | 1.362 (7) | C19—H19A | 0.9600 |
| C4—Cl1 | 1.745 (5) | C19—H19B | 0.9600 |
| C5—C6 | 1.379 (7) | C19—H19C | 0.9600 |
| C5—H5 | 0.9300 | C20—H20A | 0.9600 |
| C6—H6 | 0.9300 | C20—H20B | 0.9600 |
| C8—C9 | 1.431 (6) | C20—H20C | 0.9600 |
| C8—H8 | 0.9300 | | |

| | | | |
|---|---|---|---|
| O2—Sn1—C19 | 92.74 (19) | C10—C9—C18 | 119.8 (4) |
| O2—Sn1—C20 | 97.3 (2) | C8—C9—C18 | 118.5 (4) |
| C19—Sn1—C20 | 124.0 (3) | O2—C10—C9 | 124.2 (4) |
| O2—Sn1—O1 | 153.39 (13) | O2—C10—C11 | 116.4 (4) |
| C19—Sn1—O1 | 94.45 (19) | C9—C10—C11 | 119.4 (5) |
| C20—Sn1—O1 | 99.8 (2) | C12—C11—C10 | 121.5 (5) |
| O2—Sn1—N2 | 81.53 (13) | C12—C11—H11 | 119.2 |
| C19—Sn1—N2 | 125.1 (2) | C10—C11—H11 | 119.2 |
| C20—Sn1—N2 | 110.8 (2) | C11—C12—C13 | 121.4 (5) |
| O1—Sn1—N2 | 73.39 (13) | C11—C12—H12 | 119.3 |
| C7—O1—Sn1 | 114.3 (3) | C13—C12—H12 | 119.3 |
| C10—O2—Sn1 | 135.1 (3) | C14—C13—C18 | 120.1 (5) |
| C7—N1—N2 | 111.0 (4) | C14—C13—C12 | 121.2 (5) |
| C8—N2—N1 | 114.3 (4) | C18—C13—C12 | 118.8 (4) |
| C8—N2—Sn1 | 129.3 (3) | C15—C14—C13 | 121.6 (5) |
| N1—N2—Sn1 | 116.3 (3) | C15—C14—H14 | 119.2 |
| C6—C1—C2 | 118.2 (4) | C13—C14—H14 | 119.2 |
| C6—C1—C7 | 120.1 (4) | C14—C15—C16 | 119.3 (5) |
| C2—C1—C7 | 121.7 (4) | C14—C15—H15 | 120.4 |
| C3—C2—C1 | 121.1 (4) | C16—C15—H15 | 120.4 |
| C3—C2—H2 | 119.4 | C17—C16—C15 | 120.6 (5) |
| C1—C2—H2 | 119.4 | C17—C16—H16 | 119.7 |
| C2—C3—C4 | 118.9 (5) | C15—C16—H16 | 119.7 |
| C2—C3—H3 | 120.5 | C16—C17—C18 | 121.7 (5) |
| C4—C3—H3 | 120.5 | C16—C17—H17 | 119.1 |
| C5—C4—C3 | 121.2 (5) | C18—C17—H17 | 119.1 |
| C5—C4—Cl1 | 119.8 (4) | C17—C18—C13 | 116.7 (4) |
| C3—C4—Cl1 | 118.9 (4) | C17—C18—C9 | 124.2 (4) |
| C4—C5—C6 | 120.1 (5) | C13—C18—C9 | 119.1 (4) |
| C4—C5—H5 | 120.0 | Sn1—C19—H19A | 109.5 |
| C6—C5—H5 | 120.0 | Sn1—C19—H19B | 109.5 |
| C5—C6—C1 | 120.5 (5) | H19A—C19—H19B | 109.5 |
| C5—C6—H6 | 119.7 | Sn1—C19—H19C | 109.5 |
| C1—C6—H6 | 119.7 | H19A—C19—H19C | 109.5 |
| O1—C7—N1 | 124.8 (5) | H19B—C19—H19C | 109.5 |
| O1—C7—C1 | 118.5 (4) | Sn1—C20—H20A | 109.5 |
| N1—C7—C1 | 116.7 (4) | Sn1—C20—H20B | 109.5 |
| N2—C8—C9 | 127.5 (4) | H20A—C20—H20B | 109.5 |
| N2—C8—H8 | 116.3 | Sn1—C20—H20C | 109.5 |

| | | | |
|---|---|---|---|
| C9—C8—H8 | 116.3 | H20A—C20—H20C | 109.5 |
| C10—C9—C8 | 121.6 (4) | H20B—C20—H20C | 109.5 |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C5—H5···O2$^{i}$ | 0.93 | 2.54 | 3.388 (6) | 152 |

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