

Dichlorido[4-cyclohexyl-1-[1-(2-pyridyl- κN)ethylidene]thiosemicarbazidato- $\kappa^2 N^1, S$]phenyltin(IV)

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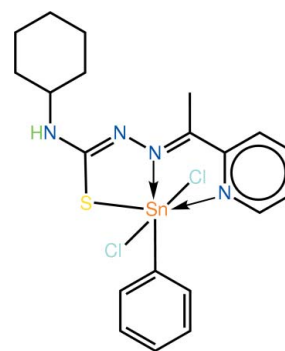
Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(C-C) = 0.003$ Å; R factor = 0.018; wR factor = 0.047; data-to-parameter ratio = 15.2.

The Sn^{IV} atom in the title compound, [Sn(C₆H₅)(C₁₄H₁₉N₄S)Cl₂], exists within a distorted octahedral geometry defined by the *N,N',S*-tridentate monodeprotonated Schiff base ligand, two mutually *trans* Cl atoms, and the *ipso-C* atom of the Sn-bound phenyl group; the latter is *trans* to the azo-N atom. The greatest distortion from the ideal geometry is found in the nominally *trans* angle formed by the S and pyridyl-N atoms at Sn [151.03 (4)°]. With the exception of the cyclohexyl group (chair form), the Schiff base ligand is almost planar (r.m.s. deviation of non-H and Sn atoms = 0.053 Å). The nearly orthogonal orientation of the Sn-bound phenyl group [N–Sn–C–C torsion angle = 70.8 (5)°] to the planar portion of the Schiff base allows for the formation of significant intramolecular C–H...Cl interactions which preclude the Cl atoms from participating in N–H...Cl hydrogen bonds. Instead, C–H... π contacts, involving methylene H and the Sn-bound phenyl group, lead to the formation of supramolecular chains that pack in the *bc* plane. Connections between these layers are of the type C–H...Cl.

Related literature

For the structure of the methyltin derivative, see: Salam *et al.* (2010).

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Experimental

Crystal data

[Sn(C₆H₅)(C₁₄H₁₉N₄S)Cl₂]
 $M_r = 542.08$
Monoclinic, $P2_1/n$
 $a = 11.5213$ (2) Å
 $b = 13.3795$ (2) Å
 $c = 15.2648$ (2) Å
 $\beta = 109.630$ (2)°

$V = 2216.30$ (6) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 1.50$ mm⁻¹
 $T = 150$ K
 $0.37 \times 0.21 \times 0.15$ mm

Data collection

Area diffractometer
Absorption correction: multi-scan
(*CrysAlis RED*; Oxford Diffraction, 2002)
 $T_{\min} = 0.730$, $T_{\max} = 0.798$

23984 measured reflections
3900 independent reflections
3642 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.018$
 $wR(F^2) = 0.047$
 $S = 1.02$
3900 reflections
257 parameters
1 restraint

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.30$ e Å⁻³
 $\Delta\rho_{\min} = -0.45$ e Å⁻³

Table 1

Selected bond lengths (Å).

| | | | |
|--------|------------|--------|-------------|
| Sn–Cl1 | 2.4587 (5) | Sn–N1 | 2.2552 (15) |
| Sn–Cl2 | 2.5083 (5) | Sn–N2 | 2.2309 (15) |
| Sn–S1 | 2.4768 (5) | Sn–C15 | 2.1552 (17) |

Table 2

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the C15–C20 benzene ring.

| <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> |
|------------------------------|-------------|---------------|-----------------------|-------------------------|
| C11–H11B...Cg1 ⁱ | 0.99 | 2.60 | 3.522 (2) | 155 |
| C13–H13B...Cl1 ⁱⁱ | 0.99 | 2.80 | 3.632 (2) | 142 |
| C16–H16...Cl2 | 0.95 | 2.66 | 3.350 (2) | 130 |
| C20–H20...Cl1 | 0.95 | 2.70 | 3.363 (2) | 127 |

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2002); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2002); data reduction: *CrysAlis RED*; 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, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5715).

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supporting information

Acta Cryst. (2010). E66, m1503–m1504 [https://doi.org/10.1107/S1600536810044247]

Dichlorido{4-cyclohexyl-1-[1-(2-pyridyl- κ N)ethylidene]thiosemicarbazidato- κ^2 N¹,S}phenyltin(IV)

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S1. Comment

The title compound, (I), is the phenyltin derivative of the recently described structure of dichlorido{4-cyclohexyl-1-[1-(2-pyridyl- κ N)ethylidene]thiosemicarbazidato- κ^2 N¹,S}methyltin(IV) (Salam *et al.*, 2010).

The Sn atom in (I), Fig. 1, exists within a six atom CCl₂N₂S donor set defined by the tridentate monodeprotonated Schiff base ligand, two mutually *trans* chlorido atoms, and the *ispo*-C atom of the Sn-bound phenyl group which is *trans* to the azo-N atom, Table 1. Distortions from the ideal octahedral geometry are ascribed primarily to the restricted bite distances formed by the Schiff base which results in an angle of 151.03 (4) ° for the nominally *trans* S1—Sn—N1 angle. The disposition of donor atoms resembles that found in the structure of the methyltin derivative (Salam *et al.*, 2010). With the exception of the cyclohexyl group, which adopts a chair conformation, the Schiff base ligand is planar. Thus, the r.m.s. deviation from the least-squares plane through the 15 non-H atoms in the conjugated part of the ligand including the Sn and methine-C15 atoms, *i.e.* Sn,S1,N1–N4,C1–C9, is 0.053 Å. The Sn-bound phenyl group occupies a position almost orthogonal to the chelate rings as seen in the N2—Sn—C15—C16 torsion angle of 70.8 (5) Å.

In the crystal, supramolecular chains along the *b* axis are mediated by C—H \cdots π interactions involving a methylene-H interacting with the Sn-bound phenyl group, Table 1. The chains pack in the *bc* plane and connections between the layers stacked along the *a* axis are of the type C—H \cdots Cl, Fig. 2 and Table 1. The non-participation of the N—H atom in forming a significant intermolecular interaction contrasts the formation of N—H \cdots Cl interactions in the methyltin derivative (Salam *et al.*, 2010). It is noted that the orthogonal orientation of the Sn-bound phenyl group allows for the formation of close intramolecular C—H \cdots Cl contacts, Table 1, which probably deactivate the chlorido atoms from forming significant hydrogen bonds. Further, it is noted that the C—H \cdots π interactions present in (I) involves the Sn-bound phenyl group as the acceptor, and that these are not possible in the methyltin derivative. Together, these factors explain the absence of significant hydrogen bonding interactions involving the N—H atom.

S2. Experimental

2-Acetylpyridine-*N*-cyclohexyl thiosemicarbazone (0.28 g, 1.0 mmol) was dissolved in absolute methanol (10 ml) in a Schlenk round bottom flask under a nitrogen atmosphere and stirred for 30 min. Then, a 10 ml methanolic solution of phenyltin(IV) trichloride (0.302 g, 1.0 mmol) was added drop wise while stirring which resulted in the formation of a yellow solution. The reaction mixture was refluxed for 4 h. and then cooled to room temperature. Yellow micro crystals of (I) were obtained from the slow evaporation of the solution at room temperature. The micro crystals were filtered off, washed with a small amount of cool methanol and dried *in vacuo* over silica gel. Light-yellow crystals were obtained from the slow evaporation of a chloroform/methanol (1/1) solution of (I) held at room temperature. Yield: 0.45 g, 77%:

M.pt.: 523–525 K. Anal. Calc. for $C_{20}H_{24}Cl_2N_4SSn$: C, 44.30; H, 4.46; N, 10.33%. Found: C, 44.12; H, 4.27; N, 10.18%.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions ($C-H = 0.95$ to 1.00 Å) and were included in the refinement in the riding model approximation, with $U_{iso}(H)$ set to $1.2-1.5U_{equiv}(C)$. The N-bound H atom was located from a difference map and refined with the distance restraint $N-H = 0.88 \pm 0.01$ Å, and with $U_{iso}(H) = 1.2U_{eq}(N)$.

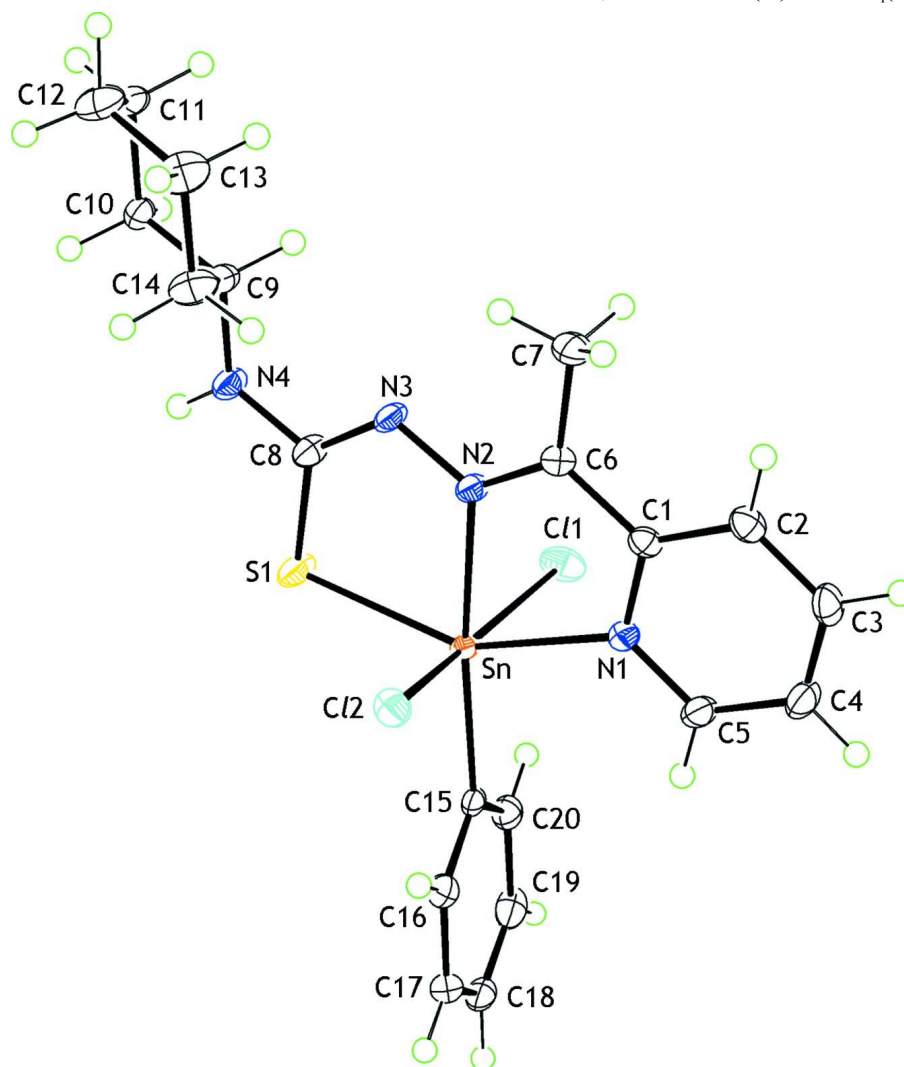


Figure 1

The molecular structure of (I) showing displacement ellipsoids at the 50% probability level.

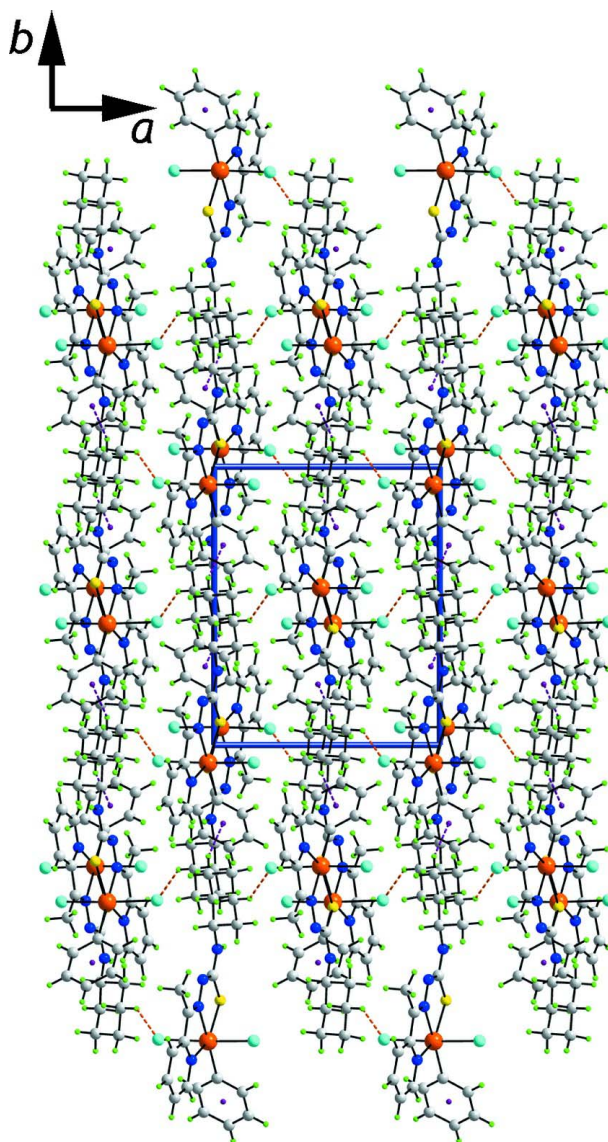


Figure 2

Unit-cell contents shown in projection down the c axis in (I). The C–H \cdots Cl and C–H \cdots π contacts are shown as orange and purple dashed lines, respectively.

Dichlorido[4-cyclohexyl-1-[1-(2-pyridyl)- κ N]ethylidene]thiosemicarbazidato- κ^2N^1,S]phenyltin(IV)

Crystal data

[Sn(C₆H₅)(C₁₄H₁₉N₄S)Cl₂]

$M_r = 542.08$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 11.5213 (2) \text{ \AA}$

$b = 13.3795 (2) \text{ \AA}$

$c = 15.2648 (2) \text{ \AA}$

$\beta = 109.630 (2)^\circ$

$V = 2216.30 (6) \text{ \AA}^3$

$Z = 4$

$F(000) = 1088$

$D_x = 1.625 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 17836 reflections

$\theta = 2.4\text{--}28.9^\circ$

$\mu = 1.50 \text{ mm}^{-1}$

$T = 150 \text{ K}$

Prism, orange

$0.37 \times 0.21 \times 0.15 \text{ mm}$

Data collection

| | |
|---|--|
| Area | 3900 independent reflections |
| diffractometer | 3642 reflections with $I > 2\sigma(I)$ |
| Graphite monochromator | $R_{\text{int}} = 0.034$ |
| $\omega/2\theta$ scans | $\theta_{\text{max}} = 25.0^\circ$, $\theta_{\text{min}} = 2.4^\circ$ |
| Absorption correction: multi-scan | $h = -13 \rightarrow 13$ |
| (<i>CrysAlis RED</i> ; Oxford Diffraction, 2002) | $k = -15 \rightarrow 15$ |
| $T_{\text{min}} = 0.730$, $T_{\text{max}} = 0.798$ | $l = -18 \rightarrow 18$ |
| 23984 measured reflections | |

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.018$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.047$ | $w = 1/[\sigma^2(F_o^2) + (0.0232P)^2 + 1.3698P]$ |
| $S = 1.02$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 3900 reflections | $(\Delta/\sigma)_{\text{max}} = 0.003$ |
| 257 parameters | $\Delta\rho_{\text{max}} = 0.30 \text{ e } \text{\AA}^{-3}$ |
| 1 restraint | $\Delta\rho_{\text{min}} = -0.45 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | |

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|--------------|--------------|----------------------------------|
| Sn | 0.473542 (11) | 0.564452 (9) | 0.716818 (8) | 0.01429 (6) |
| Cl1 | 0.25305 (5) | 0.55811 (4) | 0.62427 (4) | 0.02697 (12) |
| Cl2 | 0.67571 (4) | 0.56343 (3) | 0.84773 (3) | 0.02149 (11) |
| S1 | 0.52690 (6) | 0.41751 (4) | 0.63924 (4) | 0.02665 (13) |
| N1 | 0.39962 (14) | 0.63184 (11) | 0.82288 (10) | 0.0162 (3) |
| N2 | 0.43068 (15) | 0.43990 (10) | 0.79821 (11) | 0.0163 (3) |
| N3 | 0.45002 (15) | 0.34299 (11) | 0.78079 (11) | 0.0186 (3) |
| N4 | 0.51413 (16) | 0.23328 (12) | 0.69436 (11) | 0.0214 (4) |
| H4N | 0.5435 (19) | 0.2243 (16) | 0.6499 (11) | 0.026* |
| C1 | 0.36701 (17) | 0.56662 (13) | 0.87825 (13) | 0.0160 (4) |
| C2 | 0.31498 (17) | 0.60120 (15) | 0.94234 (13) | 0.0198 (4) |
| H2 | 0.2945 | 0.5557 | 0.9827 | 0.024* |
| C3 | 0.29312 (18) | 0.70261 (15) | 0.94712 (13) | 0.0230 (4) |
| H3 | 0.2559 | 0.7269 | 0.9898 | 0.028* |
| C4 | 0.32570 (19) | 0.76806 (15) | 0.88952 (14) | 0.0242 (4) |
| H4 | 0.3110 | 0.8377 | 0.8916 | 0.029* |

| | | | | |
|------|--------------|---------------|--------------|------------|
| C5 | 0.38022 (18) | 0.72975 (14) | 0.82871 (13) | 0.0203 (4) |
| H5 | 0.4047 | 0.7744 | 0.7898 | 0.024* |
| C6 | 0.38569 (17) | 0.46014 (14) | 0.86329 (13) | 0.0166 (4) |
| C7 | 0.3507 (2) | 0.38016 (14) | 0.91856 (14) | 0.0240 (4) |
| H7A | 0.2608 | 0.3740 | 0.8978 | 0.036* |
| H7B | 0.3817 | 0.3978 | 0.9847 | 0.036* |
| H7C | 0.3867 | 0.3164 | 0.9093 | 0.036* |
| C8 | 0.49310 (18) | 0.32842 (14) | 0.71163 (12) | 0.0193 (4) |
| C9 | 0.49837 (18) | 0.14827 (13) | 0.75032 (13) | 0.0186 (4) |
| H9 | 0.4180 | 0.1560 | 0.7613 | 0.022* |
| C10 | 0.49480 (18) | 0.05184 (13) | 0.69666 (14) | 0.0179 (4) |
| H10A | 0.5731 | 0.0436 | 0.6841 | 0.022* |
| H10B | 0.4268 | 0.0548 | 0.6363 | 0.022* |
| C11 | 0.47560 (19) | -0.03703 (14) | 0.75323 (14) | 0.0213 (4) |
| H11A | 0.3950 | -0.0304 | 0.7626 | 0.026* |
| H11B | 0.4745 | -0.0996 | 0.7184 | 0.026* |
| C12 | 0.5780 (2) | -0.04246 (15) | 0.84755 (15) | 0.0301 (5) |
| H12A | 0.6577 | -0.0555 | 0.8383 | 0.036* |
| H12B | 0.5615 | -0.0985 | 0.8841 | 0.036* |
| C13 | 0.5861 (2) | 0.05525 (15) | 0.90122 (15) | 0.0305 (5) |
| H13A | 0.5103 | 0.0639 | 0.9173 | 0.037* |
| H13B | 0.6571 | 0.0522 | 0.9599 | 0.037* |
| C14 | 0.6013 (2) | 0.14412 (15) | 0.84392 (14) | 0.0272 (5) |
| H14A | 0.6011 | 0.2066 | 0.8786 | 0.033* |
| H14B | 0.6816 | 0.1392 | 0.8339 | 0.033* |
| C15 | 0.51354 (17) | 0.69707 (13) | 0.65203 (12) | 0.0151 (4) |
| C16 | 0.62574 (18) | 0.74626 (14) | 0.68942 (14) | 0.0204 (4) |
| H16 | 0.6849 | 0.7224 | 0.7453 | 0.024* |
| C17 | 0.65198 (19) | 0.83006 (14) | 0.64572 (15) | 0.0242 (4) |
| H17 | 0.7292 | 0.8628 | 0.6714 | 0.029* |
| C18 | 0.5663 (2) | 0.86581 (14) | 0.56524 (14) | 0.0245 (5) |
| H18 | 0.5845 | 0.9231 | 0.5355 | 0.029* |
| C19 | 0.45379 (19) | 0.81846 (15) | 0.52775 (13) | 0.0242 (4) |
| H19 | 0.3943 | 0.8439 | 0.4728 | 0.029* |
| C20 | 0.42738 (18) | 0.73337 (14) | 0.57055 (13) | 0.0199 (4) |
| H20 | 0.3506 | 0.7002 | 0.5441 | 0.024* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| Sn | 0.01933 (9) | 0.01020 (8) | 0.01541 (8) | -0.00154 (5) | 0.00855 (6) | -0.00036 (4) |
| Cl1 | 0.0224 (3) | 0.0325 (3) | 0.0231 (3) | -0.0101 (2) | 0.0038 (2) | -0.0018 (2) |
| Cl2 | 0.0195 (2) | 0.0233 (3) | 0.0212 (2) | 0.00270 (18) | 0.00619 (19) | 0.00428 (18) |
| S1 | 0.0507 (4) | 0.0124 (2) | 0.0274 (3) | -0.0002 (2) | 0.0270 (3) | -0.0005 (2) |
| N1 | 0.0174 (8) | 0.0144 (8) | 0.0174 (8) | -0.0014 (6) | 0.0068 (6) | -0.0016 (6) |
| N2 | 0.0219 (9) | 0.0122 (8) | 0.0161 (8) | -0.0020 (6) | 0.0081 (7) | -0.0011 (6) |
| N3 | 0.0283 (9) | 0.0094 (7) | 0.0211 (8) | -0.0005 (7) | 0.0122 (7) | -0.0015 (6) |
| N4 | 0.0340 (10) | 0.0133 (8) | 0.0220 (9) | 0.0000 (7) | 0.0163 (8) | -0.0012 (7) |

| | | | | | | |
|-----|-------------|-------------|-------------|-------------|-------------|-------------|
| C1 | 0.0135 (9) | 0.0182 (10) | 0.0153 (9) | -0.0033 (7) | 0.0037 (7) | -0.0016 (7) |
| C2 | 0.0200 (10) | 0.0242 (10) | 0.0162 (9) | -0.0029 (8) | 0.0074 (8) | -0.0020 (8) |
| C3 | 0.0213 (10) | 0.0274 (11) | 0.0226 (10) | -0.0012 (9) | 0.0102 (8) | -0.0098 (8) |
| C4 | 0.0271 (11) | 0.0163 (10) | 0.0290 (11) | 0.0008 (8) | 0.0094 (9) | -0.0064 (8) |
| C5 | 0.0240 (10) | 0.0144 (9) | 0.0228 (10) | -0.0025 (8) | 0.0081 (8) | -0.0021 (8) |
| C6 | 0.0173 (9) | 0.0166 (9) | 0.0154 (9) | -0.0020 (8) | 0.0048 (8) | 0.0004 (7) |
| C7 | 0.0318 (11) | 0.0207 (10) | 0.0248 (10) | -0.0027 (9) | 0.0166 (9) | 0.0026 (8) |
| C8 | 0.0255 (10) | 0.0139 (9) | 0.0184 (9) | 0.0000 (8) | 0.0072 (8) | -0.0010 (7) |
| C9 | 0.0241 (10) | 0.0132 (9) | 0.0199 (9) | -0.0015 (8) | 0.0094 (8) | 0.0003 (8) |
| C10 | 0.0208 (10) | 0.0145 (9) | 0.0198 (10) | 0.0009 (8) | 0.0085 (8) | -0.0012 (7) |
| C11 | 0.0271 (11) | 0.0128 (9) | 0.0270 (11) | -0.0035 (8) | 0.0129 (9) | -0.0024 (8) |
| C12 | 0.0369 (13) | 0.0184 (10) | 0.0322 (12) | -0.0001 (9) | 0.0080 (10) | 0.0088 (9) |
| C13 | 0.0373 (13) | 0.0275 (12) | 0.0212 (11) | -0.0025 (9) | 0.0024 (10) | 0.0039 (9) |
| C14 | 0.0309 (11) | 0.0200 (10) | 0.0261 (11) | -0.0066 (9) | 0.0035 (9) | -0.0006 (8) |
| C15 | 0.0206 (9) | 0.0108 (8) | 0.0183 (9) | 0.0021 (7) | 0.0121 (8) | -0.0005 (7) |
| C16 | 0.0197 (10) | 0.0174 (9) | 0.0256 (10) | 0.0028 (8) | 0.0096 (8) | 0.0025 (8) |
| C17 | 0.0246 (10) | 0.0153 (9) | 0.0380 (12) | -0.0016 (8) | 0.0173 (9) | 0.0002 (9) |
| C18 | 0.0379 (12) | 0.0142 (9) | 0.0322 (11) | 0.0037 (9) | 0.0261 (10) | 0.0057 (8) |
| C19 | 0.0347 (12) | 0.0226 (10) | 0.0197 (10) | 0.0100 (9) | 0.0150 (9) | 0.0064 (8) |
| C20 | 0.0227 (10) | 0.0194 (10) | 0.0196 (9) | 0.0017 (8) | 0.0096 (8) | -0.0005 (8) |

Geometric parameters (Å, °)

| | | | |
|--------|-------------|----------|-----------|
| Sn—C11 | 2.4587 (5) | C9—C10 | 1.521 (2) |
| Sn—C12 | 2.5083 (5) | C9—C14 | 1.520 (3) |
| Sn—S1 | 2.4768 (5) | C9—H9 | 1.0000 |
| Sn—N1 | 2.2552 (15) | C10—C11 | 1.529 (3) |
| Sn—N2 | 2.2309 (15) | C10—H10A | 0.9900 |
| Sn—C15 | 2.1552 (17) | C10—H10B | 0.9900 |
| S1—C8 | 1.7558 (19) | C11—C12 | 1.526 (3) |
| N1—C5 | 1.337 (2) | C11—H11A | 0.9900 |
| N1—C1 | 1.353 (2) | C11—H11B | 0.9900 |
| N2—C6 | 1.294 (2) | C12—C13 | 1.529 (3) |
| N2—N3 | 1.357 (2) | C12—H12A | 0.9900 |
| N3—C8 | 1.323 (2) | C12—H12B | 0.9900 |
| N4—C8 | 1.338 (2) | C13—C14 | 1.520 (3) |
| N4—C9 | 1.470 (2) | C13—H13A | 0.9900 |
| N4—H4N | 0.863 (19) | C13—H13B | 0.9900 |
| C1—C2 | 1.387 (3) | C14—H14A | 0.9900 |
| C1—C6 | 1.470 (3) | C14—H14B | 0.9900 |
| C2—C3 | 1.386 (3) | C15—C16 | 1.391 (3) |
| C2—H2 | 0.9500 | C15—C20 | 1.392 (3) |
| C3—C4 | 1.379 (3) | C16—C17 | 1.389 (3) |
| C3—H3 | 0.9500 | C16—H16 | 0.9500 |
| C4—C5 | 1.382 (3) | C17—C18 | 1.378 (3) |
| C4—H4 | 0.9500 | C17—H17 | 0.9500 |
| C5—H5 | 0.9500 | C18—C19 | 1.382 (3) |
| C6—C7 | 1.499 (3) | C18—H18 | 0.9500 |

| | | | |
|------------|--------------|---------------|-------------|
| C7—H7A | 0.9800 | C19—C20 | 1.396 (3) |
| C7—H7B | 0.9800 | C19—H19 | 0.9500 |
| C7—H7C | 0.9800 | C20—H20 | 0.9500 |
| C15—Sn—N2 | 172.75 (6) | N4—C9—C14 | 111.40 (16) |
| C15—Sn—N1 | 101.00 (6) | C10—C9—C14 | 110.28 (16) |
| N2—Sn—N1 | 72.01 (5) | N4—C9—H9 | 108.6 |
| C15—Sn—Cl1 | 95.95 (5) | C10—C9—H9 | 108.6 |
| N2—Sn—Cl1 | 85.12 (4) | C14—C9—H9 | 108.6 |
| N1—Sn—Cl1 | 81.94 (4) | C9—C10—C11 | 109.78 (16) |
| C15—Sn—S1 | 107.97 (5) | C9—C10—H10A | 109.7 |
| N2—Sn—S1 | 79.07 (4) | C11—C10—H10A | 109.7 |
| N1—Sn—S1 | 151.03 (4) | C9—C10—H10B | 109.7 |
| Cl1—Sn—S1 | 94.152 (19) | C11—C10—H10B | 109.7 |
| C15—Sn—Cl2 | 94.91 (5) | H10A—C10—H10B | 108.2 |
| N2—Sn—Cl2 | 82.66 (4) | C12—C11—C10 | 110.94 (16) |
| N1—Sn—Cl2 | 84.48 (4) | C12—C11—H11A | 109.5 |
| Cl1—Sn—Cl2 | 164.001 (18) | C10—C11—H11A | 109.5 |
| S1—Sn—Cl2 | 93.632 (18) | C12—C11—H11B | 109.5 |
| C8—S1—Sn | 95.44 (6) | C10—C11—H11B | 109.5 |
| C5—N1—C1 | 120.18 (16) | H11A—C11—H11B | 108.0 |
| C5—N1—Sn | 123.46 (13) | C11—C12—C13 | 110.71 (17) |
| C1—N1—Sn | 116.22 (11) | C11—C12—H12A | 109.5 |
| C6—N2—N3 | 119.00 (15) | C13—C12—H12A | 109.5 |
| C6—N2—Sn | 119.47 (12) | C11—C12—H12B | 109.5 |
| N3—N2—Sn | 121.53 (11) | C13—C12—H12B | 109.5 |
| C8—N3—N2 | 115.31 (15) | H12A—C12—H12B | 108.1 |
| C8—N4—C9 | 123.75 (16) | C14—C13—C12 | 111.01 (18) |
| C8—N4—H4N | 115.6 (15) | C14—C13—H13A | 109.4 |
| C9—N4—H4N | 120.5 (14) | C12—C13—H13A | 109.4 |
| N1—C1—C2 | 120.13 (16) | C14—C13—H13B | 109.4 |
| N1—C1—C6 | 116.24 (16) | C12—C13—H13B | 109.4 |
| C2—C1—C6 | 123.59 (17) | H13A—C13—H13B | 108.0 |
| C1—C2—C3 | 119.52 (18) | C9—C14—C13 | 111.42 (16) |
| C1—C2—H2 | 120.2 | C9—C14—H14A | 109.3 |
| C3—C2—H2 | 120.2 | C13—C14—H14A | 109.3 |
| C4—C3—C2 | 119.64 (18) | C9—C14—H14B | 109.3 |
| C4—C3—H3 | 120.2 | C13—C14—H14B | 109.3 |
| C2—C3—H3 | 120.2 | H14A—C14—H14B | 108.0 |
| C3—C4—C5 | 118.37 (18) | C16—C15—C20 | 119.14 (17) |
| C3—C4—H4 | 120.8 | C16—C15—Sn | 120.92 (13) |
| C5—C4—H4 | 120.8 | C20—C15—Sn | 119.93 (13) |
| N1—C5—C4 | 122.13 (18) | C15—C16—C17 | 120.47 (18) |
| N1—C5—H5 | 118.9 | C15—C16—H16 | 119.8 |
| C4—C5—H5 | 118.9 | C17—C16—H16 | 119.8 |
| N2—C6—C1 | 116.06 (16) | C18—C17—C16 | 120.14 (19) |
| N2—C6—C7 | 122.37 (17) | C18—C17—H17 | 119.9 |
| C1—C6—C7 | 121.55 (17) | C16—C17—H17 | 119.9 |

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|--------------|--------------|-----------------|--------------|
| C6—C7—H7A | 109.5 | C17—C18—C19 | 120.10 (18) |
| C6—C7—H7B | 109.5 | C17—C18—H18 | 119.9 |
| H7A—C7—H7B | 109.5 | C19—C18—H18 | 119.9 |
| C6—C7—H7C | 109.5 | C18—C19—C20 | 120.10 (18) |
| H7A—C7—H7C | 109.5 | C18—C19—H19 | 120.0 |
| H7B—C7—H7C | 109.5 | C20—C19—H19 | 120.0 |
| N3—C8—N4 | 116.01 (17) | C15—C20—C19 | 120.03 (18) |
| N3—C8—S1 | 128.61 (14) | C15—C20—H20 | 120.0 |
| N4—C8—S1 | 115.38 (14) | C19—C20—H20 | 120.0 |
| N4—C9—C10 | 109.25 (15) | | |
| | | | |
| C15—Sn—S1—C8 | 176.61 (8) | N3—N2—C6—C7 | -1.8 (3) |
| N2—Sn—S1—C8 | -1.55 (8) | Sn—N2—C6—C7 | 177.87 (14) |
| N1—Sn—S1—C8 | -4.91 (11) | N1—C1—C6—N2 | -0.4 (3) |
| Cl1—Sn—S1—C8 | -85.76 (7) | C2—C1—C6—N2 | 177.22 (17) |
| Cl2—Sn—S1—C8 | 80.26 (7) | N1—C1—C6—C7 | -178.61 (17) |
| C15—Sn—N1—C5 | 5.52 (16) | C2—C1—C6—C7 | -1.0 (3) |
| N2—Sn—N1—C5 | -176.48 (16) | N2—N3—C8—N4 | 179.18 (16) |
| Cl1—Sn—N1—C5 | -89.02 (15) | N2—N3—C8—S1 | -0.7 (3) |
| S1—Sn—N1—C5 | -173.00 (11) | C9—N4—C8—N3 | -3.4 (3) |
| Cl2—Sn—N1—C5 | 99.47 (15) | C9—N4—C8—S1 | 176.47 (14) |
| C15—Sn—N1—C1 | -178.78 (13) | Sn—S1—C8—N3 | 1.86 (19) |
| N2—Sn—N1—C1 | -0.78 (12) | Sn—S1—C8—N4 | -177.98 (14) |
| Cl1—Sn—N1—C1 | 86.68 (12) | C8—N4—C9—C10 | 164.24 (18) |
| S1—Sn—N1—C1 | 2.70 (18) | C8—N4—C9—C14 | -73.7 (2) |
| Cl2—Sn—N1—C1 | -84.83 (12) | N4—C9—C10—C11 | -178.77 (16) |
| C15—Sn—N2—C6 | 16.3 (6) | C14—C9—C10—C11 | 58.5 (2) |
| N1—Sn—N2—C6 | 0.57 (14) | C9—C10—C11—C12 | -58.4 (2) |
| Cl1—Sn—N2—C6 | -82.53 (14) | C10—C11—C12—C13 | 56.4 (2) |
| S1—Sn—N2—C6 | -177.72 (15) | C11—C12—C13—C14 | -54.7 (3) |
| Cl2—Sn—N2—C6 | 87.12 (14) | N4—C9—C14—C13 | -179.10 (17) |
| C15—Sn—N2—N3 | -164.1 (4) | C10—C9—C14—C13 | -57.6 (2) |
| N1—Sn—N2—N3 | -179.81 (15) | C12—C13—C14—C9 | 55.6 (3) |
| Cl1—Sn—N2—N3 | 97.10 (13) | N2—Sn—C15—C16 | 70.8 (5) |
| S1—Sn—N2—N3 | 1.90 (13) | N1—Sn—C15—C16 | 86.01 (15) |
| Cl2—Sn—N2—N3 | -93.26 (13) | Cl1—Sn—C15—C16 | 168.92 (14) |
| C6—N2—N3—C8 | 178.28 (17) | S1—Sn—C15—C16 | -94.74 (14) |
| Sn—N2—N3—C8 | -1.3 (2) | Cl2—Sn—C15—C16 | 0.69 (14) |
| C5—N1—C1—C2 | -1.0 (3) | N2—Sn—C15—C20 | -110.5 (5) |
| Sn—N1—C1—C2 | -176.82 (14) | N1—Sn—C15—C20 | -95.31 (14) |
| C5—N1—C1—C6 | 176.77 (17) | Cl1—Sn—C15—C20 | -12.40 (14) |
| Sn—N1—C1—C6 | 0.9 (2) | S1—Sn—C15—C20 | 83.93 (14) |
| N1—C1—C2—C3 | 2.0 (3) | Cl2—Sn—C15—C20 | 179.37 (13) |
| C6—C1—C2—C3 | -175.53 (18) | C20—C15—C16—C17 | -0.5 (3) |
| C1—C2—C3—C4 | -1.4 (3) | Sn—C15—C16—C17 | 178.20 (14) |
| C2—C3—C4—C5 | -0.3 (3) | C15—C16—C17—C18 | 0.6 (3) |
| C1—N1—C5—C4 | -0.8 (3) | C16—C17—C18—C19 | 0.1 (3) |
| Sn—N1—C5—C4 | 174.74 (14) | C17—C18—C19—C20 | -1.0 (3) |

| | | | |
|-------------|--------------|-----------------|--------------|
| C3—C4—C5—N1 | 1.4 (3) | C16—C15—C20—C19 | -0.4 (3) |
| N3—N2—C6—C1 | -179.92 (15) | Sn—C15—C20—C19 | -179.10 (13) |
| Sn—N2—C6—C1 | -0.3 (2) | C18—C19—C20—C15 | 1.1 (3) |

Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of benzene ring C15–C20.

| <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> |
|---------------------------------------|-------------|---------------|-----------------------|-------------------------|
| C11—H11 <i>B</i> ...Cg1 ⁱ | 0.99 | 2.60 | 3.522 (2) | 155 |
| C13—H13 <i>B</i> ...C11 ⁱⁱ | 0.99 | 2.80 | 3.632 (2) | 142 |
| C16—H16...C12 | 0.95 | 2.66 | 3.350 (2) | 130 |
| C20—H20...C11 | 0.95 | 2.70 | 3.363 (2) | 127 |

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