Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

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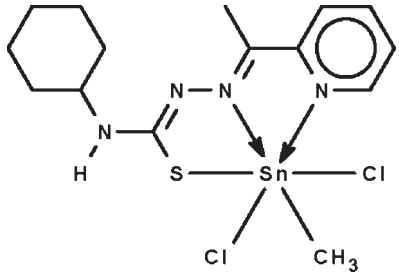
Received 19 April 2010; accepted 20 April 2010

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.021; wR factor = 0.054; data-to-parameter ratio = 21.0.

The monodeprotonated Schiff base ligand in the title compound, $[Sn(CH_3)(C_{14}H_{19}N_4S)Cl_2]$, *N*,*N*′,*S*-chelates to the Sn atom, which is six-coordinated in an octahedral environment. The three coordinating atoms along with the methyl C atom comprise a square plane, above and below which are positioned the Cl atoms. The amino group is a hydrogen-bond donor to a Cl atom of an adjacent molecule, the hydrogen bond giving rise to a helical chain extending parallel to [100].

Related literature

For the crystal structures of other metal derivatives of the Schiff base, see: Joseph *et al.* (2004); Kovala-Demertzi *et al.* (2007).



Experimental

Crystal data

$[Sn(CH_3)(C_{14}H_{19}N_4S)Cl_2]$
M_r = 480.02
Orthorhombic, $P2_12_12_1$
a = 9.2016 (5) Å
b = 12.2434 (7) Å
c = 17.4544 (10) Å
V = 1966.39 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.68 mm^{-1}
T = 293 K
0.30 × 0.25 × 0.20 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.633, T_{max} = 0.730
18838 measured reflections
4498 independent reflections
4223 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.054
S = 1.02
4498 reflections
214 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}
Absolute structure: Flack (1983), 1493 Friedel pairs
Flack parameter: −0.020 (17)

Table 1
Selected bond lengths (Å).

Sn1—C1	2.136 (3)	Sn1—S1	2.4814 (7)
Sn1—N1	2.269 (2)	Sn1—Cl1	2.4960 (7)
Sn1—N2	2.224 (2)	Sn1—Cl2	2.4701 (8)

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···Cl1^{i}	0.86 (1)	2.36 (1)	3.219 (3)	177 (3)

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank MOSTI (grant No. 06-01-09-SF0046), Universiti Malaysia Sarawak and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5250).

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supplementary materials

Acta Cryst. (2010). E**66**, m570 [**doi:10.1107/S1600536810014443**]

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

M. A. Salam, M. A. Affan, M. Shamsuddin and S. W. Ng

Comment

The mono-deprotonated anion of 2-acetylpyridine 4-cyclohexyl thiosemicarbazone is a ligand that *N*,*N*',*S*-binds to metal atoms (Joseph *et al.*, 2004). Whereas similar ligands have been complexed with diorganotin and triorganotin systems, the monoorganotin analogues have not been so extensively studied. The mono-deprotonated Schiff-base ligand in $SnCl_2(CH_3)(C_{14}H_{19}N_4S)$ *N*,*N*',*S*-chelates to the tin atom, which is six-coordinate in an octahedral environment (Scheme I, Fig. 1). The three coordinating atoms along with the methyl carbon comprise a square plane, above and below which are positioned the chlorine atoms.

Experimental

2-Acetylpyridine 4-cyclohexyl thiosemicarbazone was synthesized by using a literature method (Joseph *et al.*, 2004). The compound (0.28 g, 1 mmol) was dissolved in dry methanol (10 ml) in a Schlenk apparatus under a nitrogen atmosphere. Methyltin trichoride (0.24 g, 1 mmol) dissolved in methanol (10 ml) was added. The mixture was heated for an hour. The solvent was removed and the yellow compound recrystallized from chloroform/methanol (1:1) in 70% yield, m.p. 551-553 K.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93 to 0.978 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2 to 1.5*U*(C).

The amino H-atom was located in a difference Fourier map, and was refined isotropically with a distance restraint of N–H 0.84±0.01 Å.

Figures

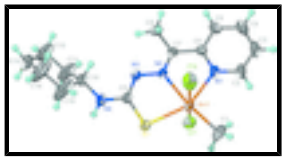


Fig. 1. Anisotropic displacement ellipsoid plot (Barbour, 2001) of $SnCl_2(CH_3)(C_{14}H_{19}N_4S)$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

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

Crystal data

$[Sn(CH_3)(C_{14}H_{19}N_4S)Cl_2]$	$F(000) = 960$
$M_r = 480.02$	$D_x = 1.621$ Mg m^{-3}

Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 8827 reflections
$a = 9.2016$ (5) Å	$\theta = 2.3$–$27.7°$
$b = 12.2434$ (7) Å	$\mu = 1.68$ mm^{-1}
$c = 17.4544$ (10) Å	$T = 293$ K
$V = 1966.39$ (19) Å^3	Prism, yellow
$Z = 4$	0.30 × 0.25 × 0.20 mm

Data collection

Bruker SMART APEX diffractometer	4498 independent reflections
Radiation source: fine-focus sealed tube	4223 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.025$
ω scans	$\theta_{max} = 27.5°$, $\theta_{min} = 2.0°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -11 \rightarrow 11$
$T_{min} = 0.633$, $T_{max} = 0.730$	$k = -15 \rightarrow 15$
18838 measured reflections	$l = -22 \rightarrow 22$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.021$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.054$	$w = 1/[\sigma^2(F_o^2) + (0.032P)^2 + 0.1524P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
4498 reflections	$\Delta\rho_{max} = 0.29$ e Å^{-3}
214 parameters	$\Delta\rho_{min} = -0.41$ e Å^{-3}
1 restraint	Absolute structure: Flack (1983), 1493 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.020 (17)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Sn1	1.061930 (18)	0.634199 (13)	0.160119 (10)	0.03748 (6)
Cl1	0.97585 (8)	0.54078 (6)	0.04139 (4)	0.04922 (17)
Cl2	1.10680 (12)	0.73379 (8)	0.28090 (5)	0.0724 (3)
S1	1.22890 (7)	0.75142 (6)	0.08461 (5)	0.04529 (16)
N1	0.8388 (3)	0.59846 (18)	0.20943 (13)	0.0411 (5)
N2	0.9157 (2)	0.77150 (16)	0.12825 (11)	0.0351 (5)
N3	0.9636 (2)	0.85860 (18)	0.08811 (12)	0.0398 (5)
N4	1.1533 (3)	0.9409 (2)	0.02881 (19)	0.0602 (7)
H4	1.2404 (17)	0.944 (3)	0.0111 (18)	0.063 (11)*

C1	1.1749 (4)	0.4916 (3)	0.1978 (2)	0.0687 (10)
H1A	1.1755	0.4893	0.2527	0.103*
H1B	1.2731	0.4936	0.1792	0.103*
H1C	1.1270	0.4277	0.1783	0.103*
C2	0.8073 (4)	0.5114 (2)	0.25205 (17)	0.0524 (7)
H2	0.8783	0.4586	0.2602	0.063*
C3	0.6713 (4)	0.4978 (3)	0.28448 (19)	0.0599 (9)
H3	0.6513	0.4370	0.3146	0.072*
C4	0.5676 (4)	0.5746 (3)	0.27171 (17)	0.0589 (8)
H4A	0.4761	0.5675	0.2938	0.071*
C5	0.5985 (3)	0.6628 (2)	0.22600 (16)	0.0490 (7)
H5	0.5271	0.7146	0.2157	0.059*
C6	0.7362 (3)	0.6743 (2)	0.19527 (14)	0.0372 (6)
C7	0.7802 (3)	0.7679 (2)	0.14765 (14)	0.0363 (5)
C8	0.6739 (3)	0.8530 (3)	0.12494 (18)	0.0517 (7)
H8A	0.7240	0.9121	0.1002	0.077*
H8B	0.6249	0.8799	0.1697	0.077*
H8C	0.6042	0.8222	0.0902	0.077*
C9	1.1021 (3)	0.8555 (2)	0.06741 (14)	0.0400 (6)
C10	1.0719 (4)	1.0403 (2)	0.0096 (2)	0.0595 (8)
H10	0.9725	1.0328	0.0290	0.071*
C11	1.1403 (6)	1.1369 (3)	0.0467 (2)	0.0941 (16)
H11A	1.2421	1.1404	0.0324	0.113*
H11B	1.1351	1.1288	0.1019	0.113*
C12	1.0651 (7)	1.2431 (3)	0.0232 (2)	0.0936 (15)
H12A	0.9671	1.2436	0.0437	0.112*
H12B	1.1171	1.3044	0.0452	0.112*
C13	1.0586 (6)	1.2563 (3)	−0.0593 (2)	0.0750 (11)
H13A	1.1561	1.2688	−0.0786	0.090*
H13B	1.0007	1.3203	−0.0712	0.090*
C14	0.9968 (8)	1.1623 (3)	−0.0982 (3)	0.123 (2)
H14A	1.0086	1.1717	−0.1531	0.147*
H14B	0.8935	1.1592	−0.0875	0.147*
C15	1.0660 (8)	1.0544 (3)	−0.0745 (2)	0.115 (2)
H15A	1.0109	0.9947	−0.0967	0.138*
H15B	1.1639	1.0509	−0.0950	0.138*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.03555 (9)	0.03065 (8)	0.04623 (9)	0.00216 (7)	−0.00566 (7)	0.00345 (7)
Cl1	0.0458 (4)	0.0469 (4)	0.0550 (4)	0.0002 (3)	−0.0045 (3)	−0.0088 (3)
Cl2	0.0986 (7)	0.0686 (5)	0.0500 (4)	−0.0184 (5)	−0.0149 (4)	−0.0065 (4)
S1	0.0341 (3)	0.0362 (3)	0.0656 (4)	0.0016 (3)	0.0043 (3)	0.0044 (3)
N1	0.0436 (13)	0.0366 (11)	0.0431 (11)	−0.0039 (10)	−0.0010 (9)	0.0059 (9)
N2	0.0395 (12)	0.0274 (10)	0.0383 (10)	0.0034 (9)	0.0030 (9)	0.0019 (8)
N3	0.0371 (11)	0.0332 (10)	0.0491 (11)	0.0028 (10)	0.0050 (9)	0.0071 (10)
N4	0.0442 (15)	0.0431 (14)	0.093 (2)	0.0067 (11)	0.0179 (14)	0.0239 (14)

C1	0.060 (2)	0.0515 (19)	0.095 (3)	0.0150 (17)	−0.021 (2)	0.0203 (18)
C2	0.061 (2)	0.0445 (16)	0.0518 (16)	−0.0074 (15)	−0.0060 (15)	0.0125 (13)
C3	0.067 (2)	0.058 (2)	0.0554 (17)	−0.0250 (18)	−0.0023 (16)	0.0152 (15)
C4	0.0554 (18)	0.067 (2)	0.0543 (16)	−0.0200 (19)	0.0087 (16)	0.0020 (14)
C5	0.0451 (17)	0.0505 (17)	0.0513 (15)	−0.0044 (12)	0.0106 (13)	−0.0029 (12)
C6	0.0389 (14)	0.0353 (13)	0.0375 (12)	−0.0017 (11)	0.0023 (11)	−0.0067 (10)
C7	0.0371 (13)	0.0348 (12)	0.0371 (13)	0.0036 (10)	0.0032 (10)	−0.0028 (10)
C8	0.0415 (15)	0.0502 (17)	0.0632 (16)	0.0141 (14)	0.0045 (13)	0.0060 (14)
C9	0.0426 (14)	0.0317 (13)	0.0458 (13)	−0.0002 (12)	0.0011 (10)	0.0017 (11)
C10	0.0435 (15)	0.0419 (15)	0.093 (2)	0.0065 (15)	0.0162 (18)	0.0256 (15)
C11	0.159 (5)	0.059 (2)	0.064 (2)	0.046 (3)	−0.036 (3)	−0.0167 (19)
C12	0.154 (4)	0.052 (2)	0.075 (2)	0.032 (3)	−0.021 (3)	−0.0103 (19)
C13	0.092 (3)	0.0485 (18)	0.085 (2)	0.013 (2)	0.013 (2)	0.0252 (17)
C14	0.214 (7)	0.071 (3)	0.083 (3)	0.057 (3)	−0.064 (4)	−0.008 (2)
C15	0.197 (6)	0.058 (2)	0.090 (3)	0.049 (3)	−0.067 (4)	−0.022 (2)

Geometric parameters (Å, °)

Sn1—C1	2.136 (3)	C5—C6	1.383 (4)
Sn1—N1	2.269 (2)	C5—H5	0.9300
Sn1—N2	2.224 (2)	C6—C7	1.473 (4)
Sn1—S1	2.4814 (7)	C7—C8	1.483 (4)
Sn1—Cl1	2.4960 (7)	C8—H8A	0.9600
Sn1—Cl2	2.4701 (8)	C8—H8B	0.9600
S1—C9	1.753 (3)	C8—H8C	0.9600
N1—C2	1.332 (4)	C10—C15	1.479 (5)
N1—C6	1.348 (3)	C10—C11	1.488 (6)
N2—C7	1.292 (3)	C10—H10	0.9800
N2—N3	1.350 (3)	C11—C12	1.529 (5)
N3—C9	1.326 (3)	C11—H11A	0.9700
N4—C9	1.330 (4)	C11—H11B	0.9700
N4—C10	1.468 (4)	C12—C13	1.450 (5)
N4—H4	0.861 (10)	C12—H12A	0.9700
C1—H1A	0.9600	C12—H12B	0.9700
C1—H1B	0.9600	C13—C14	1.453 (6)
C1—H1C	0.9600	C13—H13A	0.9700
C2—C3	1.383 (5)	C13—H13B	0.9700
C2—H2	0.9300	C14—C15	1.523 (5)
C3—C4	1.359 (5)	C14—H14A	0.9700
C3—H3	0.9300	C14—H14B	0.9700
C4—C5	1.372 (4)	C15—H15A	0.9700
C4—H4A	0.9300	C15—H15B	0.9700
C1—Sn1—N2	171.66 (12)	N2—C7—C8	122.8 (2)
C1—Sn1—N1	99.56 (12)	C6—C7—C8	121.1 (2)
N2—Sn1—N1	72.12 (8)	C7—C8—H8A	109.5
C1—Sn1—Cl2	93.43 (11)	C7—C8—H8B	109.5
N2—Sn1—Cl2	86.65 (6)	H8A—C8—H8B	109.5
N1—Sn1—Cl2	85.57 (6)	C7—C8—H8C	109.5
C1—Sn1—S1	109.56 (11)	H8A—C8—H8C	109.5

N2—Sn1—S1	78.74 (6)	H8B—C8—H8C	109.5
N1—Sn1—S1	150.85 (6)	N3—C9—N4	117.1 (3)
Cl2—Sn1—S1	93.69 (3)	N3—C9—S1	127.9 (2)
C1—Sn1—Cl1	92.01 (11)	N4—C9—S1	115.0 (2)
N2—Sn1—Cl1	86.94 (6)	N4—C10—C15	110.0 (3)
N1—Sn1—Cl1	86.53 (6)	N4—C10—C11	110.1 (3)
Cl2—Sn1—Cl1	171.05 (3)	C15—C10—C11	110.8 (3)
S1—Sn1—Cl1	91.18 (3)	N4—C10—H10	108.6
C9—S1—Sn1	95.70 (9)	C15—C10—H10	108.6
C2—N1—C6	120.1 (3)	C11—C10—H10	108.6
C2—N1—Sn1	124.3 (2)	C10—C11—C12	111.5 (3)
C6—N1—Sn1	115.58 (17)	C10—C11—H11A	109.3
C7—N2—N3	118.5 (2)	C12—C11—H11A	109.3
C7—N2—Sn1	119.51 (17)	C10—C11—H11B	109.3
N3—N2—Sn1	121.94 (16)	C12—C11—H11B	109.3
C9—N3—N2	115.7 (2)	H11A—C11—H11B	108.0
C9—N4—C10	126.0 (3)	C13—C12—C11	112.3 (3)
C9—N4—H4	123 (2)	C13—C12—H12A	109.1
C10—N4—H4	111 (2)	C11—C12—H12A	109.1
Sn1—C1—H1A	109.5	C13—C12—H12B	109.1
Sn1—C1—H1B	109.5	C11—C12—H12B	109.1
H1A—C1—H1B	109.5	H12A—C12—H12B	107.9
Sn1—C1—H1C	109.5	C12—C13—C14	113.1 (4)
H1A—C1—H1C	109.5	C12—C13—H13A	109.0
H1B—C1—H1C	109.5	C14—C13—H13A	109.0
N1—C2—C3	121.5 (3)	C12—C13—H13B	109.0
N1—C2—H2	119.3	C14—C13—H13B	109.0
C3—C2—H2	119.3	H13A—C13—H13B	107.8
C4—C3—C2	119.0 (3)	C13—C14—C15	113.3 (4)
C4—C3—H3	120.5	C13—C14—H14A	108.9
C2—C3—H3	120.5	C15—C14—H14A	108.9
C3—C4—C5	119.6 (3)	C13—C14—H14B	108.9
C3—C4—H4A	120.2	C15—C14—H14B	108.9
C5—C4—H4A	120.2	H14A—C14—H14B	107.7
C4—C5—C6	119.7 (3)	C10—C15—C14	112.7 (4)
C4—C5—H5	120.1	C10—C15—H15A	109.0
C6—C5—H5	120.1	C14—C15—H15A	109.0
N1—C6—C5	120.1 (2)	C10—C15—H15B	109.0
N1—C6—C7	116.6 (2)	C14—C15—H15B	109.0
C5—C6—C7	123.4 (2)	H15A—C15—H15B	107.8
N2—C7—C6	116.1 (2)		
C1—Sn1—S1—C9	−178.73 (15)	C3—C4—C5—C6	−1.9 (5)
N2—Sn1—S1—C9	2.07 (10)	C2—N1—C6—C5	1.0 (4)
N1—Sn1—S1—C9	3.74 (16)	Sn1—N1—C6—C5	−176.8 (2)
Cl2—Sn1—S1—C9	−83.77 (9)	C2—N1—C6—C7	180.0 (2)
Cl1—Sn1—S1—C9	88.72 (9)	Sn1—N1—C6—C7	2.1 (3)
C1—Sn1—N1—C2	2.9 (3)	C4—C5—C6—N1	0.8 (4)
N2—Sn1—N1—C2	−177.8 (2)	C4—C5—C6—C7	−178.0 (3)
Cl2—Sn1—N1—C2	−89.9 (2)	N3—N2—C7—C6	−176.5 (2)

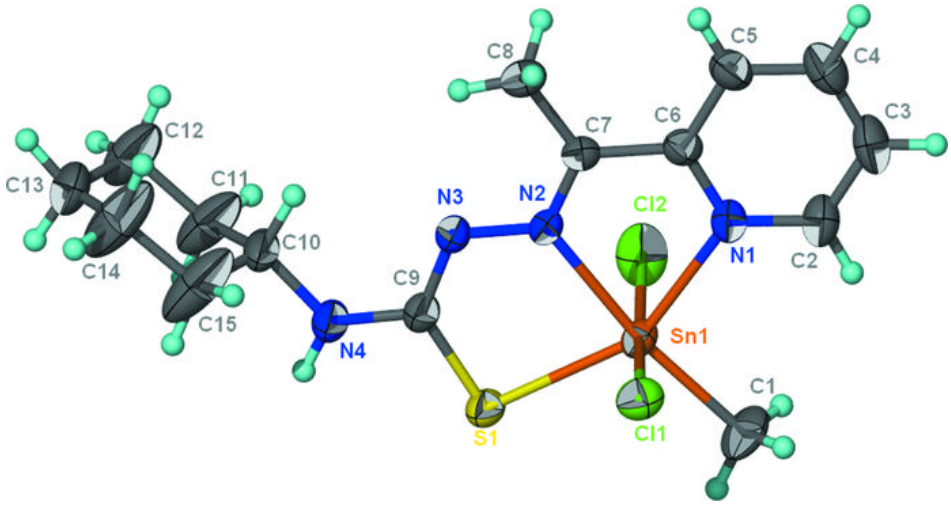
S1—Sn1—N1—C2	−179.50 (18)	Sn1—N2—C7—C6	4.4 (3)
Cl1—Sn1—N1—C2	94.3 (2)	N3—N2—C7—C8	2.6 (4)
C1—Sn1—N1—C6	−179.4 (2)	Sn1—N2—C7—C8	−176.5 (2)
N2—Sn1—N1—C6	−0.01 (17)	N1—C6—C7—N2	−4.3 (3)
Cl2—Sn1—N1—C6	87.89 (18)	C5—C6—C7—N2	174.6 (2)
S1—Sn1—N1—C6	−1.7 (3)	N1—C6—C7—C8	176.6 (2)
Cl1—Sn1—N1—C6	−87.92 (18)	C5—C6—C7—C8	−4.5 (4)
C1—Sn1—N2—C7	1.9 (9)	N2—N3—C9—N4	−179.0 (3)
N1—Sn1—N2—C7	−2.46 (18)	N2—N3—C9—S1	1.1 (3)
Cl2—Sn1—N2—C7	−88.88 (19)	C10—N4—C9—N3	2.6 (5)
S1—Sn1—N2—C7	176.68 (19)	C10—N4—C9—S1	−177.5 (3)
Cl1—Sn1—N2—C7	84.87 (19)	Sn1—S1—C9—N3	−2.6 (3)
C1—Sn1—N2—N3	−177.3 (8)	Sn1—S1—C9—N4	177.5 (2)
N1—Sn1—N2—N3	178.4 (2)	C9—N4—C10—C15	−120.0 (5)
Cl2—Sn1—N2—N3	91.99 (17)	C9—N4—C10—C11	117.6 (4)
S1—Sn1—N2—N3	−2.45 (17)	N4—C10—C11—C12	175.8 (4)
Cl1—Sn1—N2—N3	−94.26 (17)	C15—C10—C11—C12	53.9 (6)
C7—N2—N3—C9	−177.5 (2)	C10—C11—C12—C13	−54.4 (7)
Sn1—N2—N3—C9	1.6 (3)	C11—C12—C13—C14	52.4 (7)
C6—N1—C2—C3	−1.8 (4)	C12—C13—C14—C15	−50.4 (8)
Sn1—N1—C2—C3	175.8 (2)	N4—C10—C15—C14	−174.0 (5)
N1—C2—C3—C4	0.7 (5)	C11—C10—C15—C14	−52.0 (7)
C2—C3—C4—C5	1.2 (5)	C13—C14—C15—C10	50.5 (8)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4···Cl1^{i}	0.86 (1)	2.36 (1)	3.219 (3)	177 (3)

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

Fig. 1


C8
C5
C4
C12
C7
C6
C3
C13
C11
N3
N2
Cl2
C10
N1
C2
C14
C9
C15
Sn1
N4
C1
S1
Cl1