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## Structure Reports

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# Trichlorido(4-methylbenzyl)bis(1*H*-pyrazole- $\kappa$ N<sup>2</sup>)tin(IV)

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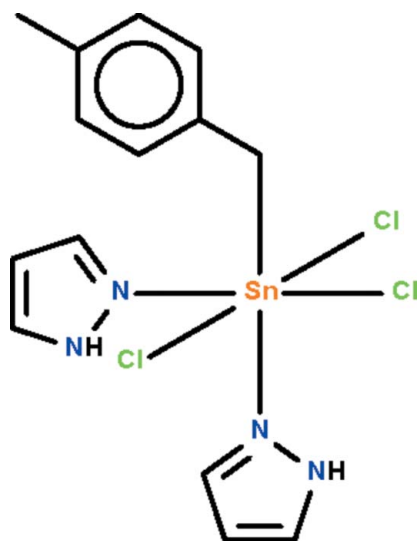
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 Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.017;  $wR$  factor = 0.044; data-to-parameter ratio = 19.5.

The six-coordinate  $\text{Sn}^{\text{IV}}$  atom in the title compound,  $[\text{Sn}(\text{C}_8\text{H}_9)\text{Cl}_3(\text{C}_3\text{H}_4\text{N}_2)_2]$ , shows an octahedral coordination. The N atoms of the N-heterocycle are *cis* to each other. The Sn–N bond that is *trans* to the Sn–C bond is shorter than the Sn–N bond *trans* to the Sn–Cl bond. Weak N–H...Cl hydrogen bonds link adjacent molecules, generating a double chain running along the *c* axis.

## Related literature

For the direct synthesis of the organotin chloride reactant, see: Sisido *et al.* (1961). For the trichloridophenyltin–di(pyrazole) adduct, see: Casas *et al.* (1996).



## Experimental

## Crystal data

$[\text{Sn}(\text{C}_8\text{H}_9)\text{Cl}_3(\text{C}_3\text{H}_4\text{N}_2)_2]$   
 $M_r = 466.36$   
 Monoclinic,  $C2/c$   
 $a = 34.7322$  (4) Å  
 $b = 7.3709$  (1) Å  
 $c = 14.5760$  (2) Å  
 $\beta = 109.0535$  (5)°

$V = 3527.13$  (8) Å<sup>3</sup>  
 $Z = 8$   
 Mo  $K\alpha$  radiation  
 $\mu = 1.90$  mm<sup>-1</sup>  
 $T = 100$  K  
 $0.30 \times 0.25 \times 0.20$  mm

## Data collection

Bruker SMART APEX  
 diffractometer  
 Absorption correction: multi-scan  
 (SADABS; Sheldrick, 1996)  
 $T_{\text{min}} = 0.599$ ,  $T_{\text{max}} = 0.702$

16131 measured reflections  
 4053 independent reflections  
 3734 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.022$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.017$   
 $wR(F^2) = 0.044$   
 $S = 1.00$   
 4053 reflections  
 208 parameters  
 2 restraints

H atoms treated by a mixture of  
 independent and constrained  
 refinement  
 $\Delta\rho_{\text{max}} = 0.43$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.26$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2}\cdots\text{Cl1}^{\text{i}}$	0.87 (1)	2.56 (2)	3.265 (2)	139 (2)
$\text{N4}-\text{H4}\cdots\text{Cl1}^{\text{ii}}$	0.88 (1)	2.65 (2)	3.270 (2)	129 (2)

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

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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5524).

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## supporting information

*Acta Cryst.* (2011). E67, m659 [doi:10.1107/S1600536811015698]

**Trichlorido(4-methylbenzyl)bis(1*H*-pyrazole- $\kappa$ N<sup>2</sup>)tin(IV)**

**Thy Chun Keng, Kong Mun Lo and Seik Weng Ng**

**S1. Comment**

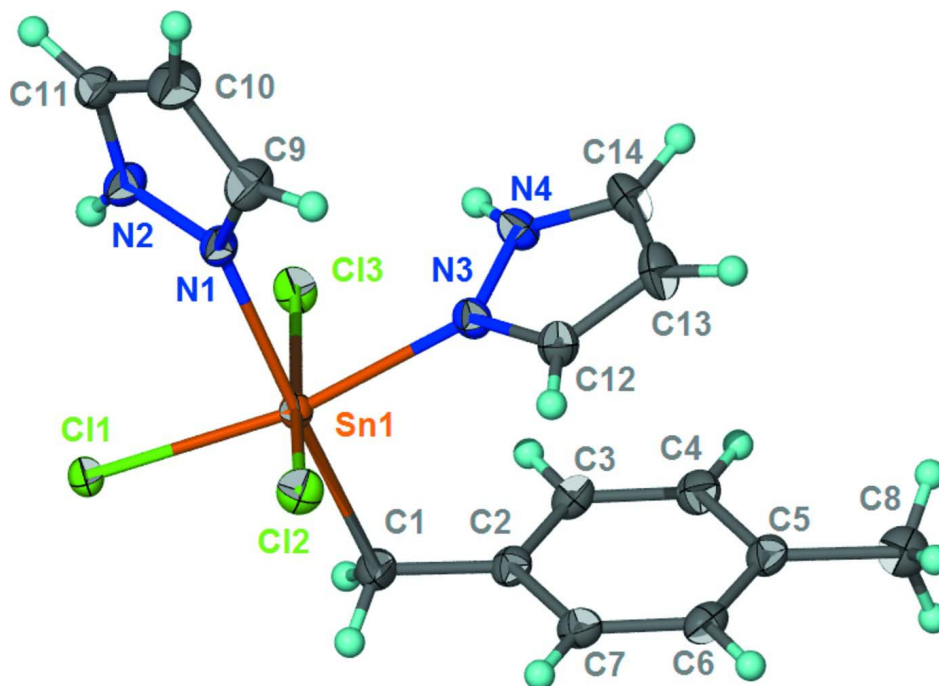
Dibenzyltin dichloride and benzyltin trichloride can be synthesized by the direct action of benzyl chloride on stannous chloride; other ring-substituted analogs are similarly synthesized (Sisido *et al.*, 1961). The title compound results from the reaction of di(4-methylbenzyl)tin dichloride with pyrazole to afford the pyrazole adduct of a monoorganotin trichloride. There are few examples of monororganotin chlorides forming adducts with *N*-heterocycles. Phenyltin trichloride forms a 1:2 adduct with pyrazole (Casas *et al.*, 1996). The 4-methylbenzyl analog affords a similar 1:2 adduct. The six-coordinate Sn<sup>IV</sup> atom in SnCl<sub>3</sub>(C<sub>8</sub>H<sub>9</sub>)(C<sub>3</sub>H<sub>4</sub>N<sub>2</sub>)<sub>2</sub> (Scheme I) shows octahedral coordination. The N atoms of the *N*-heterocycle are *cis* to each other and the three Cl atoms are coplanar (Fig. 1). The geometry can be described as being a *mer*-octahedron. The Sn–N bond that is *trans* to the Sn–C bond is shorter than the Sn–N bond *trans* to the Sn–Cl bond.

**S2. Experimental**

Di(4-methylbenzyl)tin dichloride was synthesized by using a literature procedure (Sisido *et al.*, 1961). The compound (0.4 g, 1 mmol) and pyrazole (0.136 g, 2 mmol) of pyrazole were dissolved in ethanol (100 ml) and the solution was heated for an hour. The solution was filtered and then set aside for the growth of colorless crystals.

**S3. Refinement**

Carbon-bound H-atoms were placed in calculated positions (C–H 0.95 to 0.99 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2 times *U*<sub>eq</sub>(C). The amino H-atoms were located in a difference Fourier map, and were refined isotropically with a distance restraint of N–H 0.88±0.01 Å.

**Figure 1**

Anisotropic displacement ellipsoid plot (Barbour, 2001) of  $\text{SnCl}_3(\text{C}_8\text{H}_9)(\text{C}_3\text{H}_4\text{N}_2)_2$  at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

### Trichlorido(4-methylbenzyl)bis(1H-pyrazole- $\kappa$ N<sup>2</sup>)tin(IV)

#### Crystal data

$[\text{Sn}(\text{C}_8\text{H}_9)\text{Cl}_3(\text{C}_3\text{H}_4\text{N}_2)_2]$

$M_r = 466.36$

Monoclinic,  $C2/c$

Hall symbol:  $-C\ 2yc$

$a = 34.7322\ (4)\ \text{\AA}$

$b = 7.3709\ (1)\ \text{\AA}$

$c = 14.5760\ (2)\ \text{\AA}$

$\beta = 109.0535\ (5)^\circ$

$V = 3527.13\ (8)\ \text{\AA}^3$

$Z = 8$

$F(000) = 1840$

$D_x = 1.756\ \text{Mg m}^{-3}$

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

Cell parameters from 9748 reflections

$\theta = 2.5\text{--}28.3^\circ$

$\mu = 1.90\ \text{mm}^{-1}$

$T = 100\ \text{K}$

Block, colorless

$0.30 \times 0.25 \times 0.20\ \text{mm}$

#### Data collection

Bruker SMART APEX  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega$  scans

Absorption correction: multi-scan  
(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.599$ ,  $T_{\max} = 0.702$

16131 measured reflections

4053 independent reflections

3734 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.022$

$\theta_{\max} = 27.5^\circ$ ,  $\theta_{\min} = 2.5^\circ$

$h = -44 \rightarrow 44$

$k = -9 \rightarrow 9$

$l = -18 \rightarrow 18$

Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.017$

$wR(F^2) = 0.044$

$S = 1.00$

4053 reflections

208 parameters

2 restraints

Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map

Hydrogen site location: inferred from  
neighbouring sites

H atoms treated by a mixture of independent  
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.022P)^2 + 3.9233P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.43 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.26 \text{ e } \text{\AA}^{-3}$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Sn1	0.595182 (3)	0.582348 (14)	0.721693 (7)	0.01211 (4)
Cl1	0.555981 (12)	0.42823 (5)	0.81341 (3)	0.01619 (8)
Cl2	0.626900 (12)	0.79953 (6)	0.85059 (3)	0.01902 (9)
Cl3	0.556206 (12)	0.41810 (5)	0.57311 (3)	0.01787 (9)
N1	0.54480 (4)	0.77932 (19)	0.67420 (10)	0.0150 (3)
N2	0.50505 (4)	0.7311 (2)	0.64558 (11)	0.0183 (3)
H2	0.4985 (7)	0.6177 (16)	0.6474 (18)	0.038 (7)*
N3	0.62026 (4)	0.74995 (19)	0.62547 (10)	0.0153 (3)
N4	0.60725 (4)	0.7316 (2)	0.52785 (10)	0.0166 (3)
H4	0.5873 (5)	0.657 (3)	0.4997 (15)	0.031 (6)*
C1	0.64493 (5)	0.3890 (2)	0.76417 (13)	0.0179 (3)
H1A	0.6574	0.3912	0.8357	0.021*
H1B	0.6339	0.2657	0.7453	0.021*
C2	0.67715 (5)	0.4263 (2)	0.71934 (13)	0.0161 (3)
C3	0.67158 (5)	0.3774 (2)	0.62338 (13)	0.0172 (3)
H3	0.6472	0.3172	0.5866	0.021*
C4	0.70114 (5)	0.4155 (2)	0.58085 (13)	0.0192 (4)
H4A	0.6967	0.3807	0.5154	0.023*
C5	0.73723 (5)	0.5037 (2)	0.63237 (13)	0.0179 (3)
C6	0.74257 (5)	0.5557 (2)	0.72790 (13)	0.0181 (3)
H6	0.7667	0.6179	0.7642	0.022*
C7	0.71300 (5)	0.5175 (2)	0.77065 (12)	0.0174 (3)
H7	0.7173	0.5539	0.8357	0.021*
C8	0.76965 (6)	0.5415 (3)	0.58673 (14)	0.0252 (4)
H8A	0.7843	0.6527	0.6145	0.038*
H8B	0.7569	0.5566	0.5165	0.038*
H8C	0.7888	0.4397	0.5995	0.038*
C9	0.54488 (6)	0.9595 (2)	0.66532 (15)	0.0234 (4)
H9	0.5687	1.0325	0.6800	0.028*
C10	0.50533 (6)	1.0249 (3)	0.63159 (15)	0.0255 (4)
H10	0.4970	1.1478	0.6191	0.031*
C11	0.48082 (5)	0.8757 (2)	0.62004 (12)	0.0187 (3)
H11	0.4519	0.8751	0.5979	0.022*

C12	0.65211 (5)	0.8626 (2)	0.64665 (13)	0.0177 (3)
H12	0.6676	0.9003	0.7103	0.021*
C13	0.65924 (6)	0.9165 (2)	0.56243 (14)	0.0226 (4)
H13	0.6799	0.9963	0.5572	0.027*
C14	0.63001 (6)	0.8300 (2)	0.48815 (13)	0.0215 (4)
H14	0.6266	0.8385	0.4209	0.026*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Sn1	0.01177 (6)	0.01333 (6)	0.01165 (6)	−0.00092 (4)	0.00438 (4)	−0.00068 (4)
Cl1	0.01552 (18)	0.01766 (19)	0.01712 (19)	0.00114 (14)	0.00767 (15)	0.00354 (15)
Cl2	0.01874 (19)	0.0214 (2)	0.01536 (18)	−0.00281 (15)	0.00349 (15)	−0.00550 (15)
Cl3	0.0199 (2)	0.01818 (19)	0.01524 (19)	−0.00530 (15)	0.00538 (15)	−0.00409 (15)
N1	0.0132 (6)	0.0161 (7)	0.0157 (7)	−0.0021 (5)	0.0047 (5)	−0.0007 (5)
N2	0.0138 (7)	0.0173 (7)	0.0225 (7)	−0.0023 (6)	0.0041 (6)	−0.0002 (6)
N3	0.0163 (6)	0.0172 (7)	0.0124 (6)	−0.0017 (5)	0.0048 (5)	−0.0013 (5)
N4	0.0196 (7)	0.0167 (7)	0.0134 (6)	−0.0020 (6)	0.0052 (6)	−0.0002 (6)
C1	0.0167 (8)	0.0177 (8)	0.0199 (8)	0.0016 (6)	0.0070 (7)	0.0023 (7)
C2	0.0152 (8)	0.0142 (8)	0.0194 (8)	0.0023 (6)	0.0063 (6)	0.0007 (6)
C3	0.0136 (7)	0.0172 (8)	0.0204 (8)	0.0005 (6)	0.0047 (6)	−0.0030 (7)
C4	0.0191 (8)	0.0215 (9)	0.0178 (8)	0.0032 (7)	0.0071 (7)	−0.0022 (7)
C5	0.0156 (8)	0.0167 (8)	0.0225 (9)	0.0033 (6)	0.0077 (7)	0.0029 (7)
C6	0.0137 (8)	0.0160 (8)	0.0231 (9)	0.0006 (6)	0.0039 (7)	0.0002 (7)
C7	0.0174 (8)	0.0181 (8)	0.0157 (8)	0.0022 (6)	0.0042 (6)	−0.0012 (7)
C8	0.0208 (9)	0.0311 (10)	0.0270 (10)	0.0004 (8)	0.0124 (8)	0.0025 (8)
C9	0.0198 (9)	0.0155 (8)	0.0351 (10)	−0.0020 (7)	0.0090 (8)	0.0019 (8)
C10	0.0234 (9)	0.0171 (8)	0.0358 (11)	0.0039 (7)	0.0095 (8)	0.0055 (8)
C11	0.0160 (8)	0.0226 (9)	0.0174 (8)	0.0026 (7)	0.0055 (7)	−0.0001 (7)
C12	0.0173 (8)	0.0168 (8)	0.0193 (8)	−0.0028 (6)	0.0066 (7)	−0.0010 (7)
C13	0.0270 (9)	0.0193 (9)	0.0266 (10)	−0.0049 (7)	0.0156 (8)	−0.0012 (7)
C14	0.0319 (10)	0.0176 (9)	0.0193 (8)	−0.0008 (7)	0.0141 (8)	0.0019 (7)

*Geometric parameters (Å, °)*

Sn1—C1	2.1680 (17)	C4—C5	1.394 (2)
Sn1—N1	2.2042 (14)	C4—H4A	0.9500
Sn1—N3	2.2471 (14)	C5—C6	1.397 (2)
Sn1—Cl2	2.4402 (4)	C5—C8	1.509 (2)
Sn1—Cl3	2.4646 (4)	C6—C7	1.393 (2)
Sn1—Cl1	2.4739 (4)	C6—H6	0.9500
N1—C9	1.334 (2)	C7—H7	0.9500
N1—N2	1.3528 (19)	C8—H8A	0.9800
N2—C11	1.333 (2)	C8—H8B	0.9800
N2—H2	0.869 (10)	C8—H8C	0.9800
N3—C12	1.336 (2)	C9—C10	1.386 (3)
N3—N4	1.3518 (19)	C9—H9	0.9500
N4—C14	1.336 (2)	C10—C11	1.368 (3)

N4—H4	0.876 (9)	C10—H10	0.9500
C1—C2	1.494 (2)	C11—H11	0.9500
C1—H1A	0.9900	C12—C13	1.388 (2)
C1—H1B	0.9900	C12—H12	0.9500
C2—C3	1.395 (2)	C13—C14	1.376 (3)
C2—C7	1.398 (2)	C13—H13	0.9500
C3—C4	1.390 (2)	C14—H14	0.9500
C3—H3	0.9500		
C1—Sn1—N1	178.30 (6)	C2—C3—H3	119.5
C1—Sn1—N3	96.00 (6)	C3—C4—C5	121.27 (16)
N1—Sn1—N3	82.62 (5)	C3—C4—H4A	119.4
C1—Sn1—Cl2	95.39 (5)	C5—C4—H4A	119.4
N1—Sn1—Cl2	85.54 (4)	C6—C5—C4	117.92 (16)
N3—Sn1—Cl2	87.15 (4)	C6—C5—C8	120.91 (16)
C1—Sn1—Cl3	94.93 (5)	C4—C5—C8	121.17 (16)
N1—Sn1—Cl3	84.01 (4)	C5—C6—C7	120.88 (16)
N3—Sn1—Cl3	86.29 (4)	C5—C6—H6	119.6
Cl2—Sn1—Cl3	168.293 (15)	C7—C6—H6	119.6
C1—Sn1—Cl1	94.11 (5)	C2—C7—C6	121.07 (16)
N1—Sn1—Cl1	87.23 (4)	C2—C7—H7	119.5
N3—Sn1—Cl1	169.61 (4)	C6—C7—H7	119.5
Cl2—Sn1—Cl1	94.297 (14)	C5—C8—H8A	109.5
Cl3—Sn1—Cl1	90.458 (14)	C5—C8—H8B	109.5
C9—N1—N2	105.40 (14)	H8A—C8—H8B	109.5
C9—N1—Sn1	131.25 (12)	C5—C8—H8C	109.5
N2—N1—Sn1	123.34 (11)	H8A—C8—H8C	109.5
C11—N2—N1	111.34 (14)	H8B—C8—H8C	109.5
C11—N2—H2	128.9 (16)	N1—C9—C10	110.35 (16)
N1—N2—H2	119.7 (16)	N1—C9—H9	124.8
C12—N3—N4	105.80 (13)	C10—C9—H9	124.8
C12—N3—Sn1	131.13 (11)	C11—C10—C9	105.59 (16)
N4—N3—Sn1	122.52 (10)	C11—C10—H10	127.2
C14—N4—N3	111.11 (14)	C9—C10—H10	127.2
C14—N4—H4	129.0 (15)	N2—C11—C10	107.31 (15)
N3—N4—H4	119.8 (15)	N2—C11—H11	126.3
C2—C1—Sn1	113.26 (11)	C10—C11—H11	126.3
C2—C1—H1A	108.9	N3—C12—C13	110.31 (15)
Sn1—C1—H1A	108.9	N3—C12—H12	124.8
C2—C1—H1B	108.9	C13—C12—H12	124.8
Sn1—C1—H1B	108.9	C14—C13—C12	105.33 (16)
H1A—C1—H1B	107.7	C14—C13—H13	127.3
C3—C2—C7	117.93 (15)	C12—C13—H13	127.3
C3—C2—C1	120.81 (15)	N4—C14—C13	107.45 (16)
C7—C2—C1	121.21 (15)	N4—C14—H14	126.3
C4—C3—C2	120.92 (16)	C13—C14—H14	126.3
C4—C3—H3	119.5		

N3—Sn1—N1—C9	-43.97 (16)	Cl3—Sn1—C1—C2	86.69 (12)
Cl2—Sn1—N1—C9	43.73 (16)	Cl1—Sn1—C1—C2	177.50 (12)
Cl3—Sn1—N1—C9	-130.99 (16)	Sn1—C1—C2—C3	-78.65 (18)
Cl1—Sn1—N1—C9	138.26 (16)	Sn1—C1—C2—C7	98.78 (16)
N3—Sn1—N1—N2	135.10 (13)	C7—C2—C3—C4	1.1 (2)
Cl2—Sn1—N1—N2	-137.20 (12)	C1—C2—C3—C4	178.60 (16)
Cl3—Sn1—N1—N2	48.08 (12)	C2—C3—C4—C5	-0.1 (3)
Cl1—Sn1—N1—N2	-42.67 (12)	C3—C4—C5—C6	-1.0 (3)
C9—N1—N2—C11	-0.37 (19)	C3—C4—C5—C8	178.73 (17)
Sn1—N1—N2—C11	-179.64 (11)	C4—C5—C6—C7	1.1 (2)
C1—Sn1—N3—C12	-70.61 (15)	C8—C5—C6—C7	-178.67 (17)
N1—Sn1—N3—C12	110.39 (15)	C3—C2—C7—C6	-1.0 (3)
Cl2—Sn1—N3—C12	24.52 (15)	C1—C2—C7—C6	-178.52 (16)
Cl3—Sn1—N3—C12	-165.19 (15)	C5—C6—C7—C2	-0.1 (3)
Cl1—Sn1—N3—C12	122.84 (19)	N2—N1—C9—C10	0.2 (2)
C1—Sn1—N3—N4	99.62 (13)	Sn1—N1—C9—C10	179.44 (13)
N1—Sn1—N3—N4	-79.38 (12)	N1—C9—C10—C11	0.0 (2)
Cl2—Sn1—N3—N4	-165.25 (12)	N1—N2—C11—C10	0.3 (2)
Cl3—Sn1—N3—N4	5.04 (12)	C9—C10—C11—N2	-0.2 (2)
Cl1—Sn1—N3—N4	-66.9 (3)	N4—N3—C12—C13	0.26 (19)
C12—N3—N4—C14	-0.20 (19)	Sn1—N3—C12—C13	171.71 (12)
Sn1—N3—N4—C14	-172.56 (11)	N3—C12—C13—C14	-0.2 (2)
N3—Sn1—C1—C2	-0.09 (13)	N3—N4—C14—C13	0.1 (2)
Cl2—Sn1—C1—C2	-87.78 (12)	C12—C13—C14—N4	0.1 (2)

Hydrogen-bond geometry (Å, °)

<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>
N2—H2...C11 <sup>i</sup>	0.87 (1)	2.56 (2)	3.265 (2)	139 (2)
N4—H4...C11 <sup>ii</sup>	0.88 (1)	2.65 (2)	3.270 (2)	129 (2)

Symmetry codes: (i)  $-x+1, y, -z+3/2$ ; (ii)  $x, -y+1, z-1/2$ .