Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Dichloromethyl[1-(2-pyridyl)ethanone benzoylhydrazonato]tin(IV)

M. A. Affan,[a] Mustaffa B. Shamsuddin,[b] M. Sukeri M. Yusof[c] and Bohari M. Yamin[c]*

[a]Resource Chemistry, Faculty of Resource Science and Technology, Universiti Malaysia Sarawak, 94300 Kota Samarahan, Sarawak, Malaysia, [b]Department of Chemistry, Faculty of Science, Universiti Teknologi Malaysia, 81310 UTM Skudai, Johor Darul Takzim, Malaysia, and [c]School of Chemical Sciences and Food Technology, Universiti Kebangsaan Malaysia, 43600 Bangi Selangor, Malaysia

Correspondence e-mail:
bohari@pkrisc.cc.ukm.my

The chelate Sn—O—C—N—N—C—C—N fragment, together with the substituent methyl C atom, in the title complex, $[\mathrm{SnCl_2(CH_3)(C_{14}H_{12}N_3O)}]$, is planar, allowing the tridentate *O*,*N*,*N*-donor atoms and the methyl ligand C atom to occupy the equatorial positions in an octahedral environment. The geometry of the Sn atom is distorted octahedral, with a Cl—Sn—Cl axial bond angle of 172.02 (3)°.

Received 17 November 2003
Accepted 16 December 2003
Online 24 December 2003

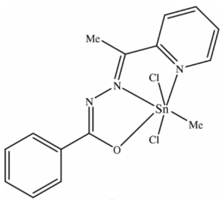
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.020
wR factor = 0.052
Data-to-parameter ratio = 19.4

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

Comment

Although the complexation of monoorganotin(IV) chloride and diorganotin chloride with benzhydrazone derivatives such as *N*-(2-pyridinylmethylene)benzhydrazone has been studied extensively (Labib *et al.*, 1996), their instability, especially that of the diorganotin(IV) complexes in common polar and non-polar organic solvents, has made it difficult to obtain good crystals for X-ray structural study. In the title compound, (I), the Sn atom is chelated by the 2-acetylpyridinebenzhydrazone ligand in a tridentate manner *via* atoms O1, N2 and N3. The coordination geometry of Sn is distorted octahedral with atoms Cl1 and Cl2 occupying the axial positions with an angle of 172.02 (3)° at the Sn atom. The equatorial atoms O1, N2, N3 and C15 make *cis* angles at the Sn atom between 73.55 (7) and 179.98 (10)°. The axial Sn1—Cl1 and Sn1—Cl2 bond distances of 2.4632 (8) and 2.4630 (8) Å, respectively, are in agreement with those in $[\mathrm{C_{22}H_{36}Cl_2N_4OSSn}]$ [2.462 (2) and 2.469 (2) Å; Carcelli *et al.*, 1995]. However, the Sn1—O1 bond distance of 2.0882 (17) Å is slightly shorter than in $[\mathrm{C_{22}H_{36}Cl_2N_4OSSn}]$ [2.108 (4) Å]. The Sn1—C15, Sn1—N2 and Sn1—N3 bond lengths are comparable with those in other organotin Schiff base complexes (Carcelli *et al.*, 1995; König *et al.*, 2000; Dey *et al.*, 2004). The ligand as a whole is not planar, the pyridine group N3/C10–C14 being at an angle of 9.18 (15)° to the C1–C6 phenyl group. The chelate fragment, together with its substituent methyl C atom and the Sn-bound methyl C atom, Sn1/01/C7/N1/N2/C8/C10/N3/C6/C9/C15 [maximum deviation of 0.080 (2) Å for O1], is essentially planar.



(I)

In the crystal structure, the molecules are linked by an intermolecular C2—H2···Cl1^{i} interaction (symmetry code as in Table 2), forming polymeric chains in a zigzag fashion parallel to the *a* axis (Fig. 2).

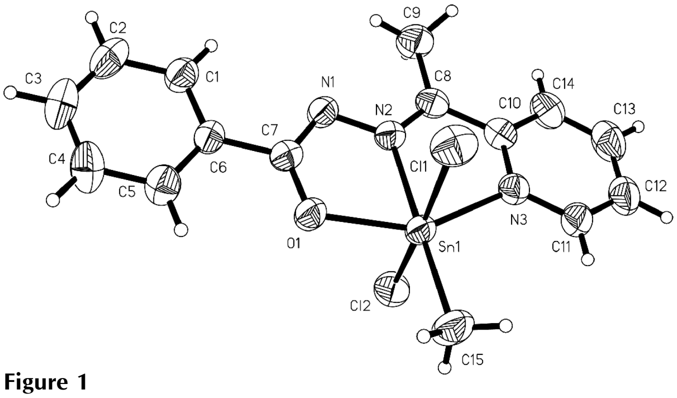


Figure 1
The molecular structure of the title compound, (I), with 50% probability displacement ellipsoids.

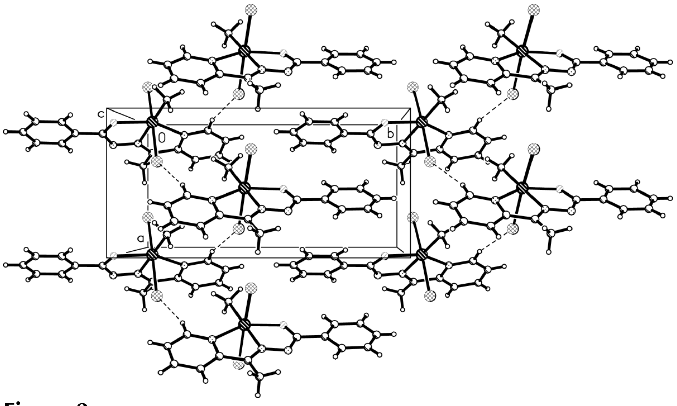


Figure 2
Packing diagram of (I), viewed down the *c* axis. Dashed lines denote C—H···Cl hydrogen bonds.

Experimental

Sodium methoxide (2.40 ml, 0.002 mol) dissolved in methanol was added to an anhydrous methanol solution containing 2,6-acetylpyridinebenzhydrazone (0.479 g, 0.002 mol) with stirring under an argon atmosphere. The colourless solution became yellow. The solution was stirred for 1 h, after which a solution of $MeSnCl_2$ (0.440 g, 0.002 mol) in methanol (10 ml) was added dropwise. The clear solution was refluxed for 4 h and then allowed to cool. The precipitated sodium chloride was removed by filtration and the filtrate was evaporated to dryness *in vacuo*. Yellow crystals were obtained from a chloroform–methanol (1:1) mixture.

Crystal data

$[SnCl_2(CH_3)(C_{14}H_{12}N_3O)]$
$M_r = 442.89$
Orthorhombic, $P2_12_12_1$
$a = 7.1865$ (11) Å
$b = 14.609$ (2) Å
$c = 16.197$ (2) Å
$V = 1700.6$ (4) Å^3
$Z = 4$
$D_x = 1.730$ Mg m^{-3}

Mo *Kα* radiation
Cell parameters from 8116 reflections
$\theta = 1.8$–27.5°
$\mu = 1.82$ mm^{-1}
$T = 293$ (2) K
Block, yellow
0.38 × 0.17 × 0.16 mm

Data collection

Bruker SMART APEX CCD area-detector
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.539$, $T_{max} = 0.751$
11 352 measured reflections

3899 independent reflections
3821 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 27.5°$
$h = -9 \rightarrow 9$
$k = -18 \rightarrow 18$
$l = -21 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.020$
$wR(F^2) = 0.052$
$S = 1.09$
3899 reflections
201 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0301P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.44$ e Å^{-3}
Absolute structure: Flack (1983), 1654 Friedel pairs
Flack parameter = 0.006 (18)

Table 1
Selected geometric parameters (Å, °).

Sn1—O1	2.0883 (16)	O1—C7	1.290 (3)
Sn1—C15	2.105 (3)	N1—C7	1.317 (3)
Sn1—N2	2.158 (2)	N1—N2	1.373 (3)
Sn1—N3	2.249 (2)	N2—C8	1.283 (3)
Sn1—Cl2	2.4630 (8)	N3—C10	1.343 (3)
Sn1—Cl1	2.4632 (8)	N3—C11	1.344 (3)
O1—Sn1—C15	106.34 (10)	N2—Sn1—Cl2	86.81 (6)
O1—Sn1—N2	73.55 (7)	N3—Sn1—Cl2	86.35 (6)
C15—Sn1—N2	179.10 (13)	O1—Sn1—Cl1	89.40 (6)
O1—Sn1—N3	145.68 (7)	C15—Sn1—Cl1	93.49 (11)
C15—Sn1—N3	107.98 (10)	N2—Sn1—Cl1	85.62 (6)
N2—Sn1—N3	72.14 (8)	N3—Sn1—Cl1	88.91 (6)
O1—Sn1—Cl2	90.89 (6)	Cl2—Sn1—Cl1	172.02 (3)
C15—Sn1—Cl2	94.08 (11)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···Cl1^i	0.93	2.77	3.587 (3)	147

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

After location in a difference Fourier map, all H atoms were positioned geometrically and treated as riding on their parent C and N atoms, with C—H = 0.93–0.96 Å and U_{iso}(H) = $1.5U_{eq}$(C) or $1.2U_{eq}$(C).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

The authors thank the Malaysian Government, Universiti-Kebangsaan Malaysia, Universiti Malaysia Sarawak and Universiti Teknologi Malaysia for research grant IRPA Nos. 09-02-02-0613 and 01/18/294/2002(32), respectively.

References

Carcelli, M., Pelizzi, C., Pelizzi, G., Mazza, P. & Zani, F. (1995). *J. Organomet. Chem.* **488**, 55–61.

Dey, D. K., Lycka, A., Mitra, S. & Rosair, G. M. (2004). *J. Organomet. Chem.* **689**, 88–95.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

König, U.-C., Berkei, M., Neikes, F., Preut, H. & Mitchell, T. N. (2000). *Acta Cryst.* C**56**, 324–326.

Labib, L., Khalil, T. E.,, Iskander, M. F. & Refaat, L. S. (1996). *Polyhedron*, **15**, 3697–3707.

Acta Cryst. (2004). E**60**, m126–m128 M. A. Affan *et al.* • $[SnCl_2(CH_3)(C_{14}H_{12}N_3O)]$ **m127**

Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

m128 M. A. Affan *et al.* • [$SnCl_2(CH_3)(C_{14}H_{12}N_3O)$] *Acta Cryst.* (2004). E**60**, m126–m128