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# BIDENTATE N, N' COORDINATION OF A PYRIDINE CARBOXALDEHYDE SCHIFF BASE LIGAND TO DIMETHYLTIN DICHLORIDE IN THE 1:1 MOLECULAR ADDUCT DICHLORODIMETHYL (N'-PHENYLPYRIDINE-2- CARBALDIMINE-N,N')TIN(IV)

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The reaction of dimethyltin(IV) dichloride with N'-phenylpyridine-2-carbaldimine results in the formation of the monomeric 1:1 complex, dichlorodimethyl(N'-phenylpyridine-2-carbaldimine-N,N')tin(IV),  $\text{Me}_2\text{SnCl}_2(\text{C}_6\text{H}_5\text{N}=\text{CHC}_5\text{H}_4\text{N}-2)$ . The ligand coordinates to the tin atom in the dichlorodimethyltin(IV) moiety through its pyridyl and imine nitrogen atoms conferring a distorted octahedral geometry to the tin atom. The two methyl groups are *trans* with respect to each other whilst the two chlorine atoms occupy *cis* positions. The bond length between the tin and the imine nitrogen atom is 2.540(5) Å whereas that of the pyridyl nitrogen atom to the tin atom is 2.439(4) Å.

KEYWORDS: dichlorodimethyl(N'-phenylpyridine-2-carbaldimine-N,N')tin(IV), dimethyltin(IV)dichloride, X-ray analysis

## INTRODUCTION

The coordinative interaction of various unsubstituted and substituted N'-phenylpyridine-2-carbaldimines with several transition metals has been extensively studied.<sup>1-7</sup> However, there have been few systematic studies carried out on the coordinating ability of the N,N bifunctional pyridine-2-carboxaldehyde Schiff base ligands with respect to organotin compounds.<sup>8-10</sup> Studies have shown that organotin compounds complexed to various N,N bifunctional ligands having Sn-N bond lengths greater than 2.39 Å exhibit anti-tumour activity.<sup>11-14</sup>

In this paper, we report the synthesis and a full X-ray structural analysis of the title compound.

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## EXPERIMENTAL

Carbon, hydrogen and nitrogen analyses were carried out on a Control Equipment Corporation 240 XA elemental analyzer at the School of Chemical Sciences, Universiti Sains Malaysia, Penang, Malaysia. Tin was estimated using an Instrumental Laboratory aa/ee 357 atomic absorption spectrophotometer. IR spectra were recorded using a Perkin Elmer FTIR 1650 spectrophotometer in the frequency range 4000–450  $\text{cm}^{-1}$ . Samples were prepared in KBr discs.  $^1\text{H}$  NMR spectra were recorded on a Brüker 300 MHz AC-P NMR spectrometer.

*N'*-phenylpyridine-2-carbaldimine

A solution of 2-pyridine carboxaldehyde (15 mmol) in 95% ethanol (10  $\text{cm}^3$ ) was added to aniline (15 mmol) in the same solvent (10  $\text{cm}^3$ ). The mixture was stirred for 1h, after which the solvent was removed. The residual liquid was distilled under low pressure to give the product. *Anal.*: Calcd. for  $\text{C}_{12}\text{H}_{10}\text{N}_2$ : C, 79.09; H, 5.53; N, 15.37%. Found: C, 79.24; H, 5.62; N, 15.60%. IR(KBr):  $\nu$ , 1620(C = N); NMR ( $^1\text{H}$ ,  $\text{CDCl}_3$ , 25°C):  $\delta$ , 8.59(s, 1H, CH = N).

*Dichlorodimethyl(N'*-phenylpyridine-2-carbaldimine-*N,N')*tin(IV)

A solution of  $\text{Me}_2\text{SnCl}_2$  (1.5mmol) in  $\text{CH}_2\text{Cl}_2$  (10  $\text{cm}^3$ ) was added to a solution of *N'*-phenylpyridine-2-carbaldimine (1.5 mmol) in a mixture of  $\text{CH}_2\text{Cl}_2$  (30  $\text{cm}^3$ ) and  $\text{CCl}_4$  (15  $\text{cm}^3$ ). The mixture was stirred for 1 h and slow evaporation at room temperature gave light yellow crystals. *Anal.*: Calcd. for  $\text{C}_{14}\text{H}_{16}\text{N}_2\text{Cl}_2\text{Sn}$ : C, 41.84; H, 4.01; N, 6.97; Sn, 29.53%. Found: C, 41.68; H, 3.85; N, 6.25; Sn, 29.67%. IR (KBr):  $\nu$ , 1600(C = N); NMR ( $^1\text{H}$ ,  $\text{CDCl}_3$ , 25°C):  $\delta$ , 8.65 (s, 1H, CH = N).

*Determination of the Crystal Structure of  $\text{C}_{14}\text{H}_{16}\text{N}_2\text{Cl}_2\text{Sn}$* 

Intensity data for a single crystal of dimensions 0.2  $\times$  0.3  $\times$  0.6 mm mounted on a thin glass fibre was collected on a Siemens  $\text{P}_4$  diffractometer fitted with graphite-monochromated  $\text{Mo-K}_\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$ . The  $\Theta$ - $2\Theta$  scan was employed to measure a total of 1921 reflections in the  $3.0^\circ \leq 2\Theta \leq 55.0^\circ$  shell. Corrections were applied for Lorentz and polarization effects but not for absorption. There were 1921 unique data of which 1675 satisfied the  $F > 4.0\sigma(F)$  criterion of observability and were used in the subsequent analysis. The structure was solved by direct methods and refined by a full-matrix least-squares procedure using Siemens SHELXTL Plus.<sup>15</sup> The crystal data and refinement parameters are given in Table 1. Fractional atomic coordinates and their equivalent isotropic displacement parameters are given in Table 2 and bond distances and angles in Table 3. The molecular structure of  $\text{C}_{14}\text{H}_{16}\text{N}_2\text{Cl}_2\text{Sn}$  with atom labelling is shown in Figure 1.

## RESULTS AND DISCUSSION

The molecular structure of the complex (Figure 1) reveals 1:1 adduct formation in which the ligand coordinates to the dichlorodimethyltin(IV) moiety *via* the pyridyl

**Table 1** Crystal data and details of structure refinement for  $(\text{CH}_3)_2\text{SnCl}_2 \cdot \text{C}_5\text{H}_4\text{NCH:NC}_6\text{H}_5$ .

Formula	$\text{C}_{14}\text{H}_{16}\text{N}_2\text{SnCl}_2$
Formula weight	401.9
Crystal System	orthorhombic
Space group	$Pna2_1$
$a(\text{\AA})$	16.153(4)
$b(\text{\AA})$	15.109(3)
$c(\text{\AA})$	6.676(1)
$V(\text{\AA}^3)$	1629.3(6)
$Z$	4
$D_c(\text{g cm}^{-3})$	1.638
$F(000)$	792
$\mu(\text{mm}^{-1})$	1.885
Data collected	1921
Unique Data	1921
Data with $F > 4.0\sigma(F)$	1675
$R$	0.028
$R_w$	0.034
$w$	$1/[\sigma^2(F_o) + 0.001F_o^2]$
$(\Delta\rho)_{\text{max}}$	$0.81\text{e}\text{\AA}^{-3}$ (near Sn atom)

**Table 2** Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement coefficients ( $\text{\AA}^2 \times 10^3$ ) with esd's in parentheses.

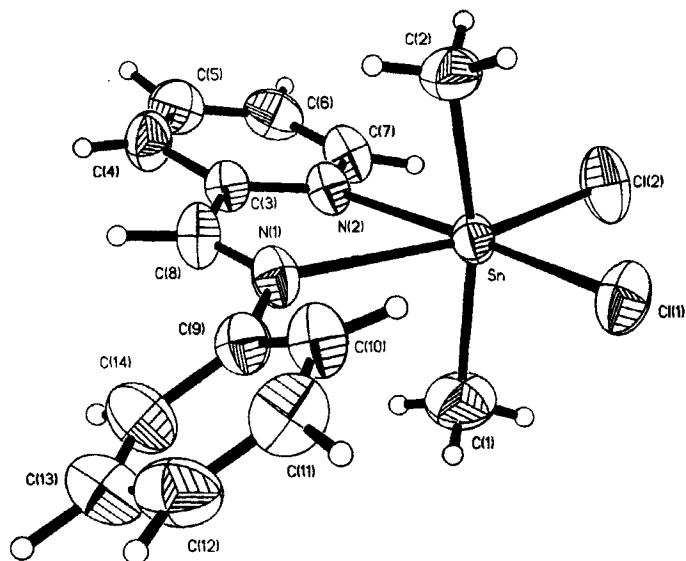
	$x/a$	$y/b$	$z/c$	$U(\text{eq})$
Sn	283(1)	2204(1)	3869	35(1)
Cl(1)	-823(1)	3049(1)	1954(3)	55(1)
Cl(2)	-661(1)	1130(1)	5442(4)	66(1)
N(1)	1636(3)	2967(3)	2991(8)	41(1)
N(2)	1461(3)	1559(3)	5627(8)	39(1)
C(1)	550(6)	1426(5)	1357(13)	68(3)
C(2)	262(4)	3129(5)	6236(11)	56(2)
C(3)	2172(3)	2014(3)	5534(9)	39(2)
C(4)	2836(4)	1812(4)	6775(11)	50(2)
C(5)	2776(4)	1099(4)	8044(11)	55(2)
C(6)	2061(4)	616(4)	8075(11)	53(2)
C(7)	1408(4)	866(4)	6848(11)	48(2)
C(8)	2245(3)	2736(3)	4067(19)	45(2)
C(9)	1757(4)	3597(3)	1458(9)	42(2)
C(10)	1127(4)	4195(4)	1012(11)	51(2)
C(11)	1223(5)	4807(4)	-485(11)	60(2)
C(12)	1942(6)	4836(5)	-1551(10)	66(3)
C(13)	2573(5)	4255(4)	-1121(19)	66(2)
C(14)	2477(5)	3638(4)	342(13)	58(2)

\*Equivalent isotropic  $U$  defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

nitrogen and the imine nitrogen atoms. The two methyl groups are *trans* with respect to each other whilst the two chlorine atoms occupy *cis* positions. The central tin atom exists in a distorted octahedral geometry; the greatest deviation from linearity in the coordination polyhedron concerns the Cl(2)-Sn-N(1) bond angle [ $158.5(1)^\circ$ ] whilst the least deviation is Cl(1)-Sn-N(2) [ $172.5(1)^\circ$ ]. The Sn-N bond lengths Sn-N(1), 2.540(5) and Sn-N(2), 2.439(4) Å [Sn-N<sub>av</sub>, 2.49 Å] are significantly greater than 2.39 Å, thus making the compound suitable for anti-tumour screening

**Table 3** Bond lengths (Å) and angles (°) with esd's in parentheses.

Sn-Cl(1)	2.541(2)	Sn-Cl(2)	2.462(2)
Sn-N(1)	2.540(5)	Sn-N(2)	2.439(4)
Sn-C(1)	2.092(8)	Sn-C(2)	2.110(8)
N(1)-C(8)	1.267(9)	N(1)-C(9)	1.411(7)
N(2)-C(3)	1.338(7)	N(2)-C(7)	1.330(8)
C(3)-C(4)	1.389(9)	C(3)-C(8)	1.471(10)
C(4)-C(5)	1.374(10)	C(5)-C(6)	1.365(10)
C(6)-C(7)	1.388(10)	C(9)-C(10)	1.394(8)
C(9)-C(14)	1.382(10)	C(10)-C(11)	1.371(9)
C(11)-C(12)	1.362(12)	C(12)-C(13)	1.376(11)
C(13)-C(14)	1.359(13)		
Cl(1)-Sn-Cl(2)	96.4(1)	Cl(1)-Sn-N(1)	105.1(1)
Cl(2)-Sn-N(1)	158.5(1)	Cl(1)-Sn-N(2)	172.5(1)
Cl(2)-Sn-N(2)	90.9(1)	N(1)-Sn-N(2)	67.7(1)
Cl(1)-Sn-C(1)	91.4(2)	Cl(2)-Sn-C(1)	95.7(2)
N(1)-Sn-C(1)	83.8(3)	N(2)-Sn-C(1)	90.1(3)
Cl(1)-Sn-C(2)	91.8(2)	Cl(2)-Sn-C(2)	96.1(2)
N(1)-Sn-C(2)	83.4(2)	N(2)-Sn-C(2)	85.2(2)
C(1)-Sn-C(2)	167.3(3)	Sn-N(1)-C(8)	114.3(4)
Sn-N(1)-C(9)	126.4(4)	C(8)-N(1)-C(9)	119.3(5)
Sn-N(2)-C(3)	116.2(3)	Sn-N(2)-C(7)	124.0(4)
C(3)-N(2)-C(7)	119.2(5)	N(2)-C(3)-C(4)	121.4(5)
N(2)-C(3)-C(8)	118.7(5)	C(4)-C(3)-C(8)	119.8(5)
C(3)-C(4)-C(5)	119.0(6)	C(4)-C(5)-C(6)	119.2(6)
C(5)-C(6)-C(7)	119.2(6)	N(2)-C(7)-C(6)	121.8(5)
N(1)-C(8)-C(3)	121.3(5)	N(1)-C(9)-C(10)	119.4(5)
N(1)-C(9)-C(14)	122.5(5)	C(10)-C(9)-C(14)	118.1(6)
C(9)-C(10)-C(11)	120.7(6)	C(10)-C(11)-C(12)	120.0(7)
C(11)-C(12)-C(13)	120.1(7)	C(12)-C(13)-C(14)	120.2(8)
C(9)-C(14)-C(13)	120.9(7)		

**Figure 1** The molecular structure of  $(\text{CH}_3)_2\text{SnCl}_2 \cdot \text{C}_3\text{H}_4\text{NCH:NC}_6\text{H}_5$ .

tests.<sup>11-14</sup> The variation in the Sn-N(1) and Sn-N(2) bond lengths (0.10Å) reflects the difference in the donor strength of the respective nitrogen atoms.

It is of interest to note that the imine nitrogen to tin bond distance observed in the six coordinate title compound [2.540(5)Å] is appreciably longer than the corresponding Sn-N distances [ranging from 2.146(2) to 2.176(4)Å] found in similar five-coordinate tin-Schiff base complexes.<sup>16-18</sup>

### Supplementary Material

Tables of hydrogen coordinates and their corresponding atomic displacement parameters, anisotropic atomic displacements for the non-hydrogen atoms and structural factors are available from the authors.

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