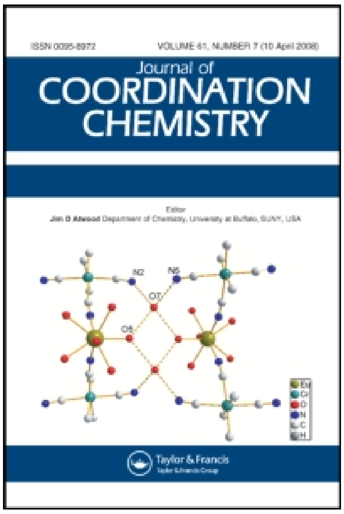

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Synthesis and Crystal Structure of Chlorotriphenyl-1- [(4'-Methoxyphenylimino)-Methyl]- 2-Naphtholtin(IV)

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SYNTHESIS AND CRYSTAL STRUCTURE OF CHLOROTRIPHENYL-1-[(4′-METHOXYPHENYLIMINO)-METHYL]-2-NAPHTHOLTIN(IV)

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The adduct derived from the reaction of 1-[(4′-methoxyphenylimino)-methyl]-2-naphthol with triphenyltin(IV) chloride, and which exhibits 1:1 stoichiometry (ligand:organotin), has been characterized spectroscopically and by an X-ray structural analysis. The complex possesses trigonal bipyramidal geometry around the Sn atom with the three phenyl rings in equatorial positions; coordination of the ligand to the metal occurs *via* the phenolic oxygen atom.

Keywords: Schiff base, chlorotriphenyltin(IV), adduct, X-ray structure

INTRODUCTION

Several reports have appeared concerning organotin and inorganic tin complexes of the Schiff base derived from the condensation of 2-hydroxy-1-naphthaldehyde with *para*-substituted aniline.[1–3] Based on infrared spectral evidence, the respective molecular adducts of tin(II) chloride and organotin(IV) chloride with the Schiff base have been deduced to involve Sn–N coordination.

However, addition compounds involving triphenyltin(IV) chloride with the Schiff base have received only scant attention. Recent studies, including an X-ray structural analysis, have shown that the interaction of Ph_3SnCl with 1-[(4′-methylphenylimino)-methyl]-2-naphthol results in a molecular adduct having a 2:3 (Ph_3SnCl : ligand) stoichiometry[3,4] and which exhibits Sn–O bonding.

In this paper we report the interaction of Ph_3SnCl with 1-[(4′-methoxyphenylimino)-methyl]-2-naphthol which leads to the formation of the title compound which crystallizes with the 1:1 (Ph_3SnCl:ligand) stoichiometry. A full X-ray structural analysis of the compound has been carried out. IR and NMR spectral data are also discussed.

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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 Instrumentation Laboratory 357 atomic absorption spectrophotometer. IR spectra were recorded using a Beckman IR 4250 spectrophotometer in the range 4000–200 cm^{-1}. Samples were prepared in KBr discs. ^1H and ^{13}C NMR spectra were recorded on a Bruker 300 MHz AC-P NMR spectrometer.

1-[(4'-Methoxyphenylimino)-methyl]-2-naphthol

A solution of 2-hydroxy-1-naphthaldehyde (50 mmol) in 50 cm^3 of a mixture of CH_2Cl_2 and 95% ethanol (2:1) was added to an ethanolic solution of *p*-anisidine (50 mmol). The mixture was stirred for 20 mins, whereupon the yellow precipitate which formed was collected and recrystallized from ethanol. Yield 81%; m.p. 96–98°C. *Anal.*: Calcd. for $HOC_{10}H_6C{:}NC_6H_4OCH_3$:C, 77.96; H, 5.46; N, 5.05%. Found: C, 77.48; H, 5.42; N, 5.10%. IR(KBr): ν, 1612(C=N), 1290(C–O, naphthol); NMR (^1H, $CDCl_3$, 25°C): δ, 3.88 (s, 3H, OCH_3), 7.1–7.8 (m, 15H, aromatic), 9.2 (s, 1H, CH=N), 13.1 (s, 1H, OH); (^{13}C, $CDCl_3$, 25°C): δ, 158.5(CH : N), 169.2(C–O, naphthol).

$[(C_6H_5)_3SnCl.HOC_{10}H_6CH{:}NC_6H_4OCH_3]$

A solution of the ligand (6 mmol) in 40 cm^3 of CH_2Cl_2 was added to triphenyltin(IV) chloride (6 mmol) in 10 cm^3 of the same solvent. The mixture was stirred and, on standing at room temperature, orange crystals were formed. Yield 75%; m.p. 120–122°C. *Anal.*: Calcd. for $C_{36}H_{30}NO_2ClSn$: C, 65.24; H; 4.57; N, 2.11; Sn, 17.91%. Found: C, 65.30; H, 4.72; N, 2.08; Sn, 17.71%. IR(KBr): ν, 1612(C=N), 1300(C–O, naphthol), 525(Sn–O); NMR (^1H, $CDCl_3$, 25°C); δ 3.88 (s, 3H, OCH_3), 7.1–7.9 (m, 30H, aromatic) 9.2 (s, 1H, CH=N), 13.1 (bs, 1H, OH); (^{13}C, $CDCl_3$, 25°C): δ, 158.5 (CH : N), 170.0(C–O, naphthol).

Determination of Crystal Structure of $C_{36}H_{30}NO_2ClSn$

Intensity data for a crystal with dimensions 0.5 × 0.3 × 0.08 mm were measured at room temperature on a Huber diffractometer using MoK_α radiation, $\lambda = 0.71069$ Å. The ω scan was employed to measure a total of 7040 reflections with $\theta_{max} \leqslant 27.5°$. No significant decomposition occurred during the data collection. Corrections were applied for Lorentz and polarization effects but not for absorption. There were 7040 unique data of which 4993 satisfied the $F \geqslant 2.5\sigma(F)$ criterion of observability and were used in the subsequent analysis. Crystal data are listed in Table I.

The structure was solved by using *SHELXS86*[5] and refined by a full-matrix least-squares procedure based on F using *SHELX76*.[6] All non-hydrogen atoms were refined with anisotropic thermal parameters and hydrogen atoms were included in their idealized positions (C–H = 0.96 Å) and refined isotropically. A weighting scheme of the form $w = k/[\sigma^2(F) + gF^2]$ was used and the refinement continued to final $R = 0.039$, $R_w = 0.042$ for $k = 0.78$ and $g = 0.00217$. The analysis of variance showed no special features and the maximum residual electron density peak in the final difference map was 0.75 e$Å^{-3}$. Fractional atomic coordinates are listed in Table

II and the numbering scheme employed is shown in Figure 1 which was drawn with ORTEP.[7] The scattering factors of all the atoms were used as incorporated in *SHELX76*.

TABLE I

Crystal data and details of structure refinement for $(C_6H_5)_3SnCl.HOC_{10}H_6CH{:}NC_6H_4OCH_3$.

Formula	$C_{36}H_{30}NO_2ClSn$
Formula weight	662.78
Crystal system	monoclinic
Space group	$P2_1/c$
a(Å)	18.175(2)
b(Å)	9.394(1)
c(Å)	19.863(2)
β(°)	115.44(1)
V(Å^3)	3062.3(5)
Z	4
D_c(g cm^{-3})	1.437
F(000)	1344
μ(cm^{-1})	9.6
Data collected	7040
Unique data	7040
Data with $F > 2.5\sigma(F)$	4993
R	0.039
R_w	0.042
w	$k/[\sigma^2(F_o) + gF_o^2]$
k	0.78
g	0.00217
$(\rho)_{max}$	0.75 e Å^{-3} (near Sn atom)

RESULTS AND DISCUSSION

The ligand and adduct have sharply defined melting points. A strong band at 1612 cm^1 due to $\nu_{C=N}$ in the IR spectrum of the ligand is found to be unaltered upon complex formation. This observation implies that N atom of the Schiff base is not involved in coordination to the Sn atom. The band at 1290 cm^{-1} in the spectrum of the ligand assignable to the C–O stretching vibration[8,9] is shifted towards higher frequency (1300 cm^{-1}) in the complex, as is consistent with the coordination of the phenolic oxygen atom to the tin atom. Two bands of medium intensity which appear in the spectrum of the complex at 525 and 440 cm^{-1} may be attributed to Sn–O and Sn–Cl vibrations, respectively. A sharp singlet attributed to the azomethine proton at 9.2 ppm in the ^{1}H NMR spectrum of the complex appears at almost the same position in the spectrum of the ligand. Broadening of the phenolic peak centred at 13.1 ppm is observed, owing to the weakening of the O–H and the strengthening of the N–H bond.[10] This further supports the Sn–O coordination mode in the complex. Further evidence for the involvement of the phenolic oxygen in the coordination of the tin atom can be obtained from the ^{13}C NMR spectra in which the peak due to the C-2 carbon atom (bonded to the phenolic oxygen atom) of the ligand is shifted by 0.8 ppm upon complexation.

TABLE II

Atomic coordinates ($\times 10^4$, $\times 10^5$ for Sn) and B_{eq}(Å^2) values with esd's in parentheses; $B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3$.

	x/a	y/b	z/c	B_{eq}
Sn1	26546(1)	15644(3)	12867(1)	2.67
Cl	2880(1)	−292(1)	2282(1)	3.48
O1	2521(2)	3054(3)	300(2)	3.54
C1	1903(2)	3632(4)	−250(2)	3.22
C2	1996(2)	4290(4)	−852(2)	2.90
C3	1305(2)	5003(4)	−1436(2)	3.05
C4	542(2)	4976(4)	−1411(2)	3.58
C5	472(3)	4270(5)	−807(3)	4.21
C6	1114(3)	3630(5)	−251(3)	4.31
C7	1350(3)	5729(5)	−2039(3)	4.51
C8	675(3)	6398(6)	−2574(3)	5.24
C9	−73(3)	6359(6)	−2539(3)	5.15
C10	−138(3)	5659(5)	−1971(3)	4.50
C11	2756(2)	4222(4)	−894(2)	2.97
N1	3400(2)	3570(4)	−407(2)	3.18
C12	4170(2)	3374(4)	−423(2)	3.19
C13	4398(3)	3997(6)	−932(3)	4.30
C14	5159(3)	3764(6)	−896(3)	4.48
C15	5714(3)	2915(5)	−348(3)	3.98
C16	5485(4)	2265(8)	158(3)	6.36
C17	4712(3)	2495(7)	112(3)	5.48
O2	6493(2)	2641(4)	−244(2)	5.54
C18	6782(3)	3267(6)	−734(4)	5.39
C22	4375(2)	2748(3)	1856(2)	4.56
C23	5217(2)	2736(3)	2087(2)	6.08
C24	5597(2)	1517(3)	1987(2)	6.04
C25	5136(2)	301(3)	1656(2)	6.11
C26	4295(2)	322(3)	1426(2)	4.97
C21	3914(2)	1541(3)	1526(2)	3.05
C32	2226(2)	4668(3)	1566(1)	4.13
C33	2159(2)	5802(3)	1989(1)	4.93
C34	2256(2)	5570(3)	2717(1)	5.65
C35	2422(2)	4203(3)	3021(1)	7.15
C36	2489(2)	3069(3)	2597(1)	6.25
C31	2391(2)	3301(3)	1870(1)	3.23
C42	1788(3)	−7(5)	−222(2)	6.73
C43	1215(3)	−913(5)	−739(2)	9.72
C44	644(3)	−1609(5)	−560(2)	10.96
C45	646(3)	−1398(5)	136(2)	10.10
C46	1219(3)	−491(5)	653(2)	6.16
C41	1790(3)	205(5)	474(2)	3.70

The structure of the complex and the atom labelling scheme is shown in Fig. 1. Bond lengths and angles are listed in Table III. The tin atom possesses trigonal bipyramidal geometry with three phenyl groups occupying the equatorial positions while a Cl atom and the phenolic oxygen atom of the ligand are situated at the axial positions. The O(1)–Sn–Cl angle is 172.4(1)°, while the angles subtended by the

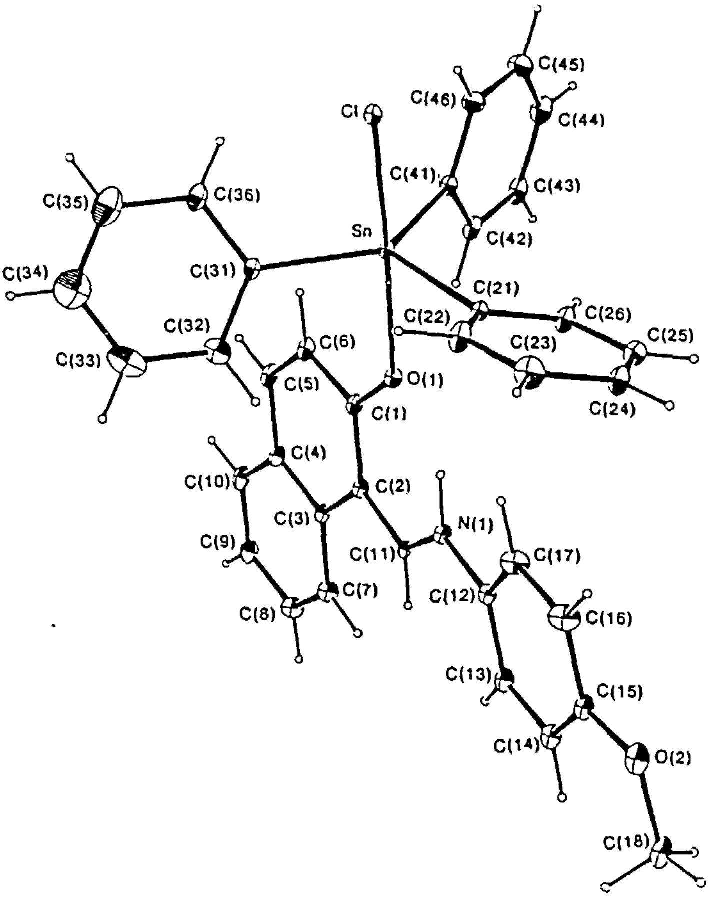

FIGURE 1 Molecular structure and atom numbering scheme employed for $(C_6H_5)_3SnCl.HOC_{10}H_6CH:NC_6H_4OCH_3$.

phenyl rings at the tin atom vary from 110.6(1) to 126.6(1)°, thus showing a slight distortion from ideal trigonal bipyramidal geometry. The Sn–Cl distance of 2.535(1) Å is within the range of Sn–Cl distances observed for other similar chloro complexes[4,11] of tin (2.336(8)–2.565(9) Å). The Sn–O distance of 2.334(3) Å is comparable with others reported earlier[3,4] (2.280(4)–2.391(3) Å).

A notable feature of the structure is that the phenolic proton has shifted from the oxygen to the imine nitrogen atom, thus effectively preventing Sn–N coordination. Therefore the ligand is coordinated to the tin atom in the form of a zwitterion. This phenomenon has been well substantiated by both Bullock *et al.*[12] and Kamwaya *et al.*[13]

TABLE III
Selected bond distances (Å) and angles (°) with esd's in parentheses.

Cl-Sn1	2.535(1)	O1-Sn1-Cl	172.4(1)
O1-Sn1	2.334(3)	C21-Sn1-Cl	91.2(1)
C21-Sn1	2.129(3)	C21-Sn1-O1	84.7(1)
C31-Sn1	2.170(3)	C31-Sn1-Cl	95.7(1)
C41-Sn1	2.128(4)	C31-Sn1-O1	91.7(1)
C1-O1	1.302(5)	C31-Sn1-C21	110.6(1)
C2-C1	1.419(5)	C41-Sn1-Cl	89.9(1)
C6-C1	1.433(6)	C41-Sn1-O1	86.9(1)
C3-C2	1.457(5)	C41-Sn1-C21	122.4(1)
C11-C2	1.421(5)	C41-Sn1-C31	126.6(1)
C4-C3	1.409(5)	C1-O1-Sn1	134.1(2)
C7-C3	1.411(5)	C2-C1-O1	121.1(3)
C5-C4	1.423(6)	C6-C1-O11	120.2(3)
C10-C4	1.413(6)	C6-C1-C2	118.8(3)
C6-C5	1.355(6)	C3-C2-C1	119.8(3)
C8-C7	1.381(6)	C11-C2-C1	120.1(3)
C9-C8	1.390(7)	C11-C2-C3	120.0(3)
C10-C9	1.353(7)	C4-C3-C2	119.2(3)
N1-C11	1.307(5)	C7-C3-C2	123.7(4)
C12-N1	1.424(5)	C7-C3-C4	117.1(4)
C13-C12	1.376(5)	C5-C4-C3	118.9(3)
C17-C12	1.373(6)	C10-C4-C3	120.3(4)
C14-C13	1.372(6)	C10-C4-C5	120.7(4)
C15-C14	1.377(6)	C6-C5-C4	122.5(4)
C16-C15	1.384(6)	C5-C6-C1	120.7(4)
O2-C15	1.364(5)	C8-C7-C3	121.1(4)
C17-C16	1.384(6)	C9-C8-C7	121.0(5)
C18-O2	1.418(6)	C10-C9-C8	119.4(4)
		C9-C10-C4	121.1(4)
		N1-C11-C2	124.2(3)
		C12-N1-C11	128.3(3)
		C13-C12-N1	124.7(4)
		C17-C12-N1	116.5(3)
		C17-C12-C13	118.8(4)
		C14-C13-C12	120.6(4)
		C15-C14-C13	120.9(4)
		C16-C15-C14	118.9(4)
		O2-C15-C14	126.1(4)
		O2-C15-C16	115.0(4)
		C17-C16-C15	119.8(5)
		C16-C17-C12	121.0(4)
		C18-O2-C15	118.9(4)
		C22-C21-Sn1	117.9(1)
		C26-C21-Sn1	121.8(1)
		C32-C31-Sn1	121.1(1)
		C36-C31-Sn1	118.4(1)
		C42-C41-Sn1	121.6(1)
		C46-C41-Sn1	118.4(1)

SUPPLEMENTARY MATERIAL AVAILABLE

Tables of fractional atomic coordinates, thermal parameters, interatomic parameters and observed and calculated structure factors are available on request from the authors.

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