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The structures of the adducts formed from organotin moieties and 1-[(4-methoxyphenylimino)methyl]-2-naphthol. Crystal structure of chloro-trimethyl[1-[(4-methoxyphenylimino)methyl]-2-naphthol]tin(IV): $Me_3SnCl \cdot [HOC_{10}H_6CH:NC_6H_4OCH_3]$

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Abstract

A series of adducts resulting from the coordinative interaction of $RSnCl_3$, R_2SnCl_2 and R_3SnCl (R = Me and Ph) with 1-[(4-methoxyphenylimino)methyl]-2-naphthol have been made. An X-ray crystal structural study of the trimethyltin(IV) derivative has been carried out, and IR, NMR, and Mössbauer data for the various are reported. All the adducts involve Sn–O coordination.

1. Introduction

The reactions of salicylideneimine and 2-hydroxy-1-naphthalideneimine with appropriate tin compounds under neutral conditions have been shown to give 1 : 1 or 1 : 2 (tin : ligand) adducts depending on the steric or electronic features of both the tin compound or the ligand [1–11]. X-ray structural studies on these adducts have revealed monodentate Sn–O coordination even though the ligand contains both a phenolic O and an imino N donor atom [2,6,8,9,11] and so is potentially bidentate. We recently investigated the electron donating effect of a methyl group on the tin atom by treating trimethyltin chloride with 1-[(4-methoxyphenylimino)methyl]-2-naphthol [10]. The adduct thus obtained exhibited 1 : 1 stoichiometry even though steric hindrance at the central tin atom is minimal. In this paper we report the synthesis and characterization of a series of adducts derived from the reactions of organotin(IV) chloride with 1-[(4-methoxyphenylimino)methyl]-2-naphthol. An x-ray structural study has been carried out on chlorotrimethyl{1-[(4-methoxyphenylimino)-methyl]-2-naphthol}tin(IV).

2. Experimental details

p-Methoxyaniline and 2-hydroxy-1-naphthaldehyde were purchased from Fluka Chemie AG. All organotin(IV) chlorides were obtained from the Aldrich Chemical Company.

Microanalyses were carried out at Universiti Sains Malaysia. The IR spectra were recorded for KBr discs on a Beckman IR 20A spectrophotometer in the frequency range 4000–250 cm^{-1}. The ^1H NMR spectra were recorded on a Bruker AC-P 300 MHz NMR spectrometer; $CDCl_3$-d_1 and DMSO-d_6 were used as the solvent, with TMS as internal standard. Mössbauer

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spectra were recorded on a constant-acceleration microprocessing spectrometer with Ca $^{119m}SnO_3$ as the source. The samples were compressed as Perspex discs and cooled to 80 K.

2.1. Synthesis of the ligand, 1-[(4-methoxyphenylimino)methyl]-2-naphthol, [$HOC_{10}H_6CH:NC_6H_4$-OCH_3] (1)

A solution of *p*-methoxyaniline (12.3 g, 0.1 mol) in a mixture of dichloromethane and 95% ethanol was added to one of 2-hydroxy-1-naphthaldehyde (17.2 g, 0.1 mol) in the same solvent mixture. The mixture was then left standing at 0°C, and a yellowish precipitate was formed. This was filtered off and recrystallized from dichloromethane; m.p. 106–108°C. Anal. Found: C, 77.21; H, 5.40; N, 5.02%. Calc. for $C_{18}H_{15}NO_2$: C, 77.95; H, 5.46; N, 5.05%. IR: ν(C=N) 1612 cm^{-1}; ν(C–O) 1290 cm^{-1}. ^{1}H NMR: δ(OH, $CDCl_3$-d_1) 13.82; δ(CH=N, $CDCl_3$-d_1) 9.40 ppm, δ(CH=N, DMSO-d_6) 9.80 ppm.

2.2. Synthesis of the adducts **2–6**

2.2.1. Trichloromethylbis{1-[(4-methoxyphenyl-imino)methyl]-2-naphthol}tin(IV), $MeSnCl_3 \cdot 2[HOC_{10}H_6$-$CH:NC_6H_4OCH_3]$ (2)

A solution of methyltin trichloride (0.72 ml, 3 mmol) in dichloromethane was treated with one of **1** (1.66 g, 6 mmol) in the same solvent. The mixture was stirred, and a yellowish solid separated; m.p. 250°C. Anal: Found: C, 55.87; H, 4.25; N, 3.32; Sn, 14.87%. Calc. for $C_{37}H_{33}N_2O_4Cl_3Sn$: C, 55.92; H, 4.19; N, 3.52; Sn, 14.94%. IR: ν(C=N) 1620 cm^{-1}; ν(C–O) 1308, 1280 cm^{-1}; ν(Sn–O) 540 cm^{-1}. ^{1}H NMR: δ(CH=N, DMSO-d_6) 9.80 ppm. Mössbauer: δ 0.91; (ΔE_Q) 2.18 mm s^{-1}.

2.2.2. Trichlorophenylbis{1-[(4-methoxyphenyl-imino)methyl]-2-naphthol}tin(IV), $PhSnCl_3 \cdot 2[HOC_{10}H_6$-$CH:NC_6H_4OCH_3]$ (3)

This complex was made in the manner described for the preparation of **2**. Phenyltin trichloride (0.5 ml, 3 mmol) was added to **1** (1.66 g, 6 mmol). The yellow to red solid separated upon stirring. M.p. 250°C. Anal: Found: C, 58.25; H, 3.95; N, 3.10; Sn, 13.66%. Calc. for $C_{42}H_{35}N_2O_4Cl_3Sn$: C, 58.88; H, 4.13; N, 3.27; Sn, 13.85%. IR: ν(C=N) 1620 cm^{-1}; ν(C–O) 1295, 1275 cm^{-1}; ν(Sn–O) 535 cm^{-1}. ^{1}H NMR: δ(CH=N, DMSO-d_6) 9.80 ppm.

2.2.3. Dichlorodimethylbis{1-[(4-methoxyphenyl-imino)methyl]-2-naphthol}tin(IV), $Me_2SnCl_2 \cdot 2[HOC_{10}$-$H_6CH:NC_6H_4OCH_3]$ (4)

A solution of **1** (1.66 g, 6 mmol) in dichloromethane was reacted with dimethyltin dichloride (0.66 g, 3 mmol) which had earlier dissolved in the same solvent. The mixture was stirred whereby the yellow to red solid was formed. M.p. 168–170°C. Anal: Found: C, 58.55; H, 4.62; N, 3.52: Sn, 15.92%. Calc. for $C_{38}H_{36}N_2O_4Ci_2Sn$: C, 58.94; H, 4.70; N, 3.62; Sn, 15.33%. IR: ν(C=N) 1620 cm^{-1}; ν(C–O) 1304, 1278 cm^{-1}; ν(Sn–O) 538 cm^{-1}. ^{1}H NMR: δ(OH, $CDCl_3$-d_1) 13.83; δ(CH=N, $CDCl_3$-d_1) 9.40 ppm. Mössbauer: δ 1.45; (ΔE_Q) 4.27 mm s^{-1}.

2.2.4. Dichlorodiphenylbis{1-[(4-methoxyphenyl-imino)methyl]-2-naphthol}tin(IV), $Ph_2SnCl_2 \cdot 2[HOC_{10}$-$H_6CH:NC_6H_4OCH_3]$ (5)

The procedure was similar with that described for preparation of **4**. Diphenyltin dichloride (1.03 g, 3 mmol) was treated with the ligand **1** (1.66 g, 6 mmol), and the yellow to red crystals were obtained upon standing at room temperature. M.p. 158–160°C. Anal: Found: C, 63.94; H, 4.43; N, 3.00; Sn, 12.96%. Calc. for $C_{48}H_{40}N_2O_4Cl_2Sn$: C, 64.17; H, 4.50; N, 3.12; Sn, 13.21%. IR: ν(C=N) 1615 cm^{-1}; ν(C–O) 1302, 1280 cm^{-1}; ν(Sn–O) 528 cm^{-1}. ^{1}H NMR: δ(OH, $CDCl_3$-d_1) 13.85; δ(CH=N, $CDCl_3$-d_1) 9.40 ppm. Mössbauer: δ 1.33; (ΔE_Q) 3.80 mm s^{-1}.

2.2.5. Chlorotrimethyl{1-[(4-methoxyphenyl-imino)methyl]-2-naphthol}tin(IV), $Me_3SnCl \cdot [HOC_{10}H_6$-$CH:NC_6H_4OCH_3]$ (6)

This synthesis was carried out by adding a solution of trimethyltin chloride (0.60 g, 3 mmol) in dichloromethane to one of **1** (1.66 g, 3 mmol) in the same solvent. The mixture was stirred under nitrogen at room temperature and then kept overnight, to give yellowish crystals. M.p. 106–108°C. Anal: Found: C, 53.07; H, 4.95; N, 2.68; Sn, 25.02%. Calc. for $C_{21}H_{24}NOClSn$: C, 52.92; H, 5.09; N, 2.94; Sn, 24.91%. IR: ν(C=N) 1620 cm^{-1}; ν(C–O) 1300, 1280 cm^{-1}; ν(Sn–O) 544 cm^{-1}. ^{1}H NMR: δ(OH, $CDCl_3$-d_1) 13.85; δ(CH=N, $CDCl_3$-d_1) 9.40 ppm. Mössbauer: δ 1.40; (ΔE_Q) 3.52 mm s^{-1}.

2.3. Crystal structure determination of $Me_3SnCl \cdot [HOC_{10}H_6CH:NC_6H_4OCH_3]$ (6)

Intensity data for a crystal with dimensions 0.5 × 0.3 × 0.2 mm^3 were measured at room temperature on a Syntex P2$_1$ diffractometer using graphite monochromated Mo Kα radiation, λ = 0.71069 Å. A $\theta/2\theta$ scan was used to give a total of 4698 reflections with $\theta_{max} \leq$ 26°. Corrections were applied for Lorentz and polarization effects but not for absorption. There were 3667 unique data, of which 3565 satisfied the $I \geq 2.5\sigma(I)$ criterion of observability and were used in the subsequent analysis. The structure was solved by using SHELXS86 [12] and refined by a full matrix least squares procedure based on F using SHELX76 [13]. All non-hy-

TABLE 1. Crystal data and details of structure refinement for $(CH_3)_3SnCl \cdot 2HOC_{10}H_6CH:NC_6H_4OCH_3$

Formula	$C_{21}H_{24}NO_2ClSn$
Formula weight	476.6
Crystal system	Monoclinic
Space group	$P2_1/n$
a (Å)	10.882(2)
b (Å)	16.332(3)
c (Å)	12.158(2)
β (°)	105.55(2)
V (Å^3)	2081(1)
Z	4
D_c (g cm^{-3})	1.521
$F(000)$	960
μ (mm^{-1})	1.370
Data collected	3698
Unique data	3667
Data with $I > 2.5\,\sigma(I)$	3565
R	0.064
R_w	0.062
w	$k/[\sigma^2(F_o) + gF_o^2]$
k	1
g	0.08
$(\Delta\rho)_{max}$ (e Å^{-3})	0.72 (near Sn atom)

drogen 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 $R = 0.064$, $R_w = 0.062$ for $k = 1$ and $g = 0.08$. The analysis of variance showed no special features and the maximum residual electron density peak in the final difference map was 0.72 e Å^{-3}. The scattering factors for all the atoms were used as incorporated in SHELX76. Crystal data and refinement parameters are summarized in Table 1. Fractional atomic coordinates are listed in Table 2 and bond lengths and angles in Table 3. Additional data, including hydrogen atom coordinates, anisotropic temperature factors, and a list of observed and calculated structure factors are available from the authors.

TABLE 2. Atomic coordinates ($\times 10^4$) and B_{eq} (Å^2) values for non-hydrogen atoms with e.s.d values in parentheses

	x	y	z	B_{eq} [a]
Sn	2122(5)	1137(1)	3814(2)	4.07
Cl(1)	2657(5)	2261(4)	5283(6)	6.88
O(1)	1542(5)	139(2)	2353(4)	3.64
C(91)	2862(6)	230(3)	5024(2)	7.98
C(92)	206(4)	1518(1)	3375(1)	6.76
C(93)	3374(6)	1552(4)	2888(6)	4.30
C(2)	182(7)	−704(3)	3060(3)	5.57
C(3)	−670(7)	−1359(3)	2910(3)	4.43
C(4)	−941(7)	−1814(3)	1903(3)	3.98
C(5)	−360(7)	−1615(3)	1046(3)	2.91
C(6)	492(7)	−960(3)	1195(3)	3.30
C(1)	763(7)	−504(3)	2202(3)	7.92
C(7)	−1856(9)	−2443(6)	1776(4)	3.86
C(8)	−2260(8)	−2913(3)	827(2)	9.86
C(9)	−1679(3)	−2766(9)	−58(9)	6.12
C(10)	−849(3)	−2105(6)	30(3)	4.90
C(11)	1176(4)	−796(8)	373(5)	1.75
C(13)	2894(4)	−631(9)	−1113(9)	3.19
C(14)	3695(4)	−444(9)	−1803(9)	3.16
C(15)	4323(4)	308(9)	−1688(11)	5.05
C(16)	4150(4)	874(9)	−883(9)	2.57
C(17)	3350(4)	688(9)	−194(11)	5.16
C(12)	2722(4)	−64(9)	−309(11)	4.34
C(18)	5694(9)	1167(1)	−2443(3)	5.13
N(1)	1973(4)	−209(3)	444(9)	3.35
O(2)	5138(3)	395(9)	−2356(6)	5.11

[a] $B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3$.

3. Result and discussion

The molecular structure of $Me_3SnCl \cdot [HOC_{10}H_6\text{-}CH:NC_6H_4OCH_3]$ (**6**), depicted in Fig. 1, shows that it is monomeric, with the phenolic oxygen atom of the ligand and the Cl atom occupying the axial positions and the three methyl groups lying in the equatorial plane.

The O–Sn–Cl angle 176.5(6)° shows a slightly distorted trigonal bipyramidal geometry around the tin atom. The C–Sn–C angles between the three methyl groups are 123.8(7), 115.0(8) and 120.7(9)°. The Sn–Cl distance 2.519(6) Å is slightly greater than that in

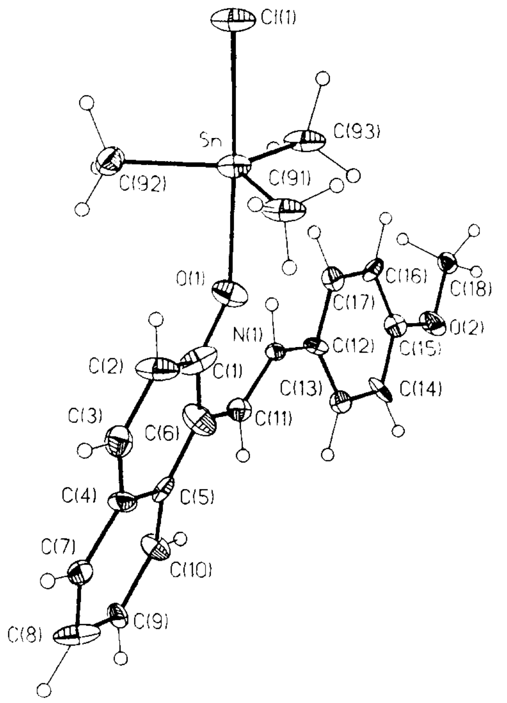

Fig 1. Molecular structure and atom numbering scheme for $Me_3SnCl \cdot [HOC_{10}H_6CH:NC_6H_4OCH_3]$.

TABLE 3. Bond distances (Å) and angles (°) with e.s.d's in parentheses

Cl(1)–Sn	2.519(6)	C(11)–C(6)	1.422(5)
O(1)–Sn	2.368(7)	C(8)–C(7)	1.357(7)
C(91)–Sn	2.092(9)	C(9)–C(8)	1.407(7)
C(92)–Sn	2.103(1)	C(10)–C(9)	1.395(7)
C(93)–Sn	2.098(1)	N(1)–C(11)	1.281(3)
C(1)–O(1)	1.331(5)	C(14)–C(13)	1.395(0)
C(3)–C(2)	1.395(0)	C(12)–C(13)	1.395(0)
C(1)–C(2)	1.395(0)	C(15)–C(14)	1.395(0)
C(4)–C(3)	1.395(0)	C(16)–C(15)	1.395(0)
C(5)–C(4)	1.395(0)	O(2)–C(15)	1.361(4)
C(7)–C(4)	1.411(6)	C(17)–C(16)	1.395(0)
C(6)–C(5)	1.395(0)	C(12)–C(17)	1.395(0)
C(10)–C(5)	1.458(3)	N(1)–C(12)	1.399(6)
C(1)–C(6)	1.395(0)	O(2)–C(18)	1.415(6)
O(1)–Sn–Cl(1)	176.5(6)	C(11)–C(6)–C(1)	119.3(7)
C(91)–Sn–Cl(1)	92.6(8)	C(2)–C(1)–O(1)	118.7(9)
C(91)–Sn–O(1)	90.9(9)	C(6)–C(1)–O(1)	121.2(8)
C(92)–Sn–Cl(1)	89.4(9)	C(6)–C(1)–C(2)	120.0(0)
C(92)–Sn–O(1)	88.1(8)	C(8)–C(7)–C(4)	124.3(6)
C(92)–Sn–C(91)	123.8(7)	C(9)–C(8)–C(7)	116.4(7)
C(93)–Sn–Cl(1)	94.9(6)	C(10)–C(9)–C(8)	119.8(8)
C(93)–Sn–O(1)	84.3(9)	C(9)–C(10)–C(5)	124.4(7)
C(93)–Sn–C(91)	115.0(8)	N(1)–C(11)–C(6)	124.5(8)
C(93)–Sn–C(92)	120.7(7)	C(12)–C(13)–C(14)	120.0(0)
C(1)–O(1)–Sn	132.7(9)	C(15)–C(14)–C(13)	120.0(0)
C(1)–C(2)–C(3)	120.0(0)	C(16)–C(15)–C(14)	120.0(0)
C(4)–C(3)–C(2)	120.0(0)	O(2)–C(15)–C(14)	114.3(8)
C(5)–C(4)–C(3)	120.0(0)	O(2)–C(15)–C(16)	125.6(9)
C(7)–C(4)–C(3)	117.5(9)	C(17)–C(16)–C(15)	120.0(0)
C(7)–C(4)–C(5)	122.4(8)	C(12)–C(17)–C(16)	120.0(0)
C(6)–C(5)–C(4)	120.0(0)	C(17)–C(12)–C(13)	120.0(0)
C(10)–C(5)–C(4)	112.1(6)	N(1)–C(12)–C(13)	124.0(9)
C(10)–C(5)–C(6)	127.6(6)	N(1)–C(12)–C(17)	116.0(9)
C(1)–C(6)–C(5)	120.0(0)	C(12)–N(1)–C(11)	125.8(7)
C(11)–C(6)–C(5)	120.5(7)	C(18)–O(2)–C(15)	119.8(7)

chlorotriphenyl{1-[(4-methylphenyl-imino)methyl]-2-naphthol}tin(IV) 2.482(2) Å [9]. This may be due to the electron releasing properties of the methyl group, which raises the electron density on the Sn atom causing elongation of the Sn–Cl bond.

A noteworthy feature of the structure is that the phenolic proton has migrated to the imine N atom, as in the case of chlorotriphenyl{1-[(4-methylphenyl-imino)methyl]-2-naphthol}tin(IV) [9] and dichloro{1-[(4-methoxyphenylimino)-methyl]-2-naphthol}diphenyltin(IV) [14].

The Mössbauer data (see experimental section) shows the δ value for **6** to be within the range 1.38–1.53 mm s^{-1} reported for similar Sn^{IV} complexes [15,16]. The large ΔE_Q value (3.52 mm s^{-1}) observed for **6** is consistent with trigonal bipyramidal structure, as proposed by Hill and coworkers [16].

The infrared spectral data show that the band at 1615 cm^{-1} characteristic of the C=N stretching vibration in the free ligand **1** is in almost the same position in the spectra of adducts **2**–**6**. This observation is consistent with the absence of coordination of the imino N atom to the tin atom. A band assignable to the C–O stretching vibration at 1290 cm^{-1} in the ligand is shifted to 1260–1280 cm^{-1}, in the adducts, and there is deformation of the O–H band at 1300 cm^{-1} [1,17] in all cases. Thus, the presence of Sn–O coordination mode, similar to that in **2**, may be inferred for adducts **3**–**6**.

In view of the differences in solubility of the adducts, the ^{1}H NMR spectra of the adducts and ligand were obtained in different solvents. Adducts **4**–**6** dissolve easily in deuterated chloroform whereas adducts **2** and **3** are only soluble in DMSO-d_6. The ^{1}H NMR spectrum for the ligand shows that the signal assigned to the azomethine proton at 9.40 ppm (in $CDCl_3$) and 9.80 ppm (in DMSO-d_6) remains unaltered in adducts **2**–**6**, further confirming the absence of coordination of the imino N atom to the tin atom.

The compounds **2**, **4**, **5** and **6** exhibit a quadrupole-split doublet in their Mössbauer spectra. Compound **2** exhibits a band at $\delta = 0.91$ mm s^{-1}, which falls within the range of 0.80–1.21 mm s^{-1} observed for similar Sn(IV) octahedral complexes [18]. The ΔE_Q for this adduct (2.18 mm s^{-1}) is also in accord with the octahedral geometry for the tin compound [18]. The δ (1.45 mm s^{-1}) and ΔE_Q (4.27 mm s^{-1}) values for **4** fall within the ranges, $\delta = 1.32$–1.59 mm s^{-1} and $\Delta E_Q = 3.80$–4.32 mm s^{-1}, respectively reported by Fitzsimmons *et al.* [19] and Barbieri *et al.* [20] for similar six-coordinate tin(IV) complexes. In the case of adduct **5**, the X-ray structure of which is known [21], the δ (1.33 mm s^{-1}) and ΔE_Q (3.80 mm s^{-1}) values are readily attributed to the six-coordinate tin(IV) compound. However, these values are lower than those observed for **4**. The smaller δ value for **5** may be ascribed to electron-withdrawal by the phenyl groups, which reduces the *s* electron density at the Sn nucleus. Parish and Platt [22] have reported that in octahedral $R_2SnX_2 \cdot 2L$ systems, the majority of *trans*-R_2 complexes have isomer shifts (δ) of > 1.2 mm s^{-1}, whereas very few *cis*-R_2 compounds exhibit isomer shifts > 1.0 mm s^{-1}. Hence, the Mössbauer data for the present adducts $R_2SnCl_2 \cdot 2L$ (R = Me, Ph) are consistent with *trans* R_2 configuration, as revealed in the crystal structure of **5** [21].

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