Journal of Organometallic Chemistry, 391 (1990) 37–46
Elsevier Sequoia S.A., Lausanne – Printed in The Netherlands
JOM 20914

Organotin(IV) complexes of some *N,N*-dimethylaniline *N*-oxide derivatives

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(Received December 13th, 1989)

Abstract

Some new organotin(IV) complexes of the general formula $R_xSnCl_{4-x} \cdot yL$, where R = Me, Ph; $x = 0$–3; $y = 1$–3; $L = R'C_6H_4NMe_2O$ (R′ = H, *o*- or *m*- or *p*-F, Cl, Br, NO_2, Me, OMe, Ph), have been prepared, mostly as white solids, and characterized by elemental analysis, and IR, ^1H, ^{13}C and ^{119}Sn NMR spectroscopy. The ligands L complex with tin through the oxygen atom of the *N*-oxide to give complexes with tin with coordination numbers of 5–7. With a few exceptions, the compounds Me_3SnCl, Ph_3SnCl and Ph_2SnCl_2 give penta-coordinate tin complexes, the compounds Me_2SnCl_2 and $PhSnCl_3$ give hexa-coordinate species, and the compound $SnCl_4$ gives $SnCl_4 \cdot 3L$ complexes containing hepta-coordinate tin.

Introduction

Organotin(IV) compounds are well known to form complexes with pyrazoles [1], pyridines [2], in which the donor site is nitrogen, and with others, such as dimethylsulphoxide (DMSO) [3] and phosphine oxides [4] in which the donor site is oxygen. However, organotin(IV) complexes containing tertiary amine *N*-oxide ligands are rather uncommon; the only previous studies that were on aromatic amine *N*-oxides [5-7]; in reference 7, we described complexes of R_2SnCl_2 (R = Me, ^nBu, Ph) with (*Z*)-*N*-benzylidene arylamine *N*-oxides (nitrones). There appear to be no previous reports on organotin(IV) complexes of aliphatic amine *N*-oxides. We thought it of interest to study therefore the products formed by reaction of the organotin (IV) compounds R_xSnCl_{4-x}, R = Me, Ph; $x = 0, 1, 2, 3$ with aliphatic amine *N*-oxides, i.e., *N,N*-dimethylaniline *N*-oxide derivatives.

Results and discussion

Organotin (IV) complexes $R_xSnCl_{4-x} \cdot yL$, where R = Me, Ph; $x = 0, 1, 2, 3$; $y = 1$ or 2 or 3; $L = R'$-$C_6H_4NMe_2O$ (R′ = H, *o*- or *m*- or *p*-F, Cl, Br, NO_2, Me,

Table 1

Physical properties and analyses for some tin complexes $R_xSnCl_{4-x}\cdot yL$.

Ligand (L)	Complex	m.p. (°C)	Analysis (found(calc.) (%))				IR data [a] (cm^{-1})			
			C	H	N	ν(N–O) (Δν)	ν(Sn–O)	ν(Sn–C)	ν(Sn–Cl)	ν(C=C)
$C_6H_5NMe_2O$	$Me_3SnCl\cdot L$	oil				970m, 920w (4), (0)	390w	545s,b	260w	1600m
	$Ph_3SnCl\cdot L$	156–158	58.95 (59.6)	5.3 (5.0)	2.5 (2.6)	980s, 925w (−6), (−5)	350m	280s	325w	1590w
	$Ph_2SnCl_2\cdot 2L$	86–88	54.9 (54.4)	5.5 (5.2)	4.6 (4.5)	945m, 920m (29), (0)	340w	290w	315w	1600w
	$PhSnCl_3\cdot 2L$	oil				993m (−19)	330s,b	280m	305s,b	1590m
	$SnCl_4\cdot 3L$	83–85	43.4 (42.9)	5.0 (4.9)	6.1 (6.2)	960m, 940w (14), (−20)	385w	–	330w	1595w
o-$MeC_6H_4NMe_2O$	$Ph_2SnCl_2\cdot L$	oil				945w (29)	335w	290w	310m	1585w
	$PhSnCl_3\cdot 2L$	oil				965m (9)	335s,b	285m	310s,b	1595m
	$SnCl_4\cdot 3L$	oil				950s (24)	330w	–	305w	1590w
m-$MeC_6H_4NMe_2O$	$Ph_3SnCl\cdot L$	101–103				985m (−10)	340m	280s	310w	1590w
	$SnCl_4\cdot 3L$	oil				940s,sh (35)	415w	–	300m	1600s,sh
p-$MeC_6H_4NMe_2O$	$Me_3SnCl\cdot L$	oil				975m (5)	392m	545w	285w	1600w
	$Ph_3SnCl\cdot L$	109–111	58.6 (60.4)	5.6 (5.2)	2.8 (2.6)	970m (10)	335w	285w	310w	1585w
	$Me_2SnCl_2\cdot 2L$	91–93				970w (10)	355w	560s	286s	1600w,b
	$Ph_2SnCl_2\cdot L$	178–180				975w (5)	350w	290w	310w	1585w
	$PhSnCl_3\cdot 2L$	oil				985m (−5)	330s,b	290m	305s,b	1600m

	$SnCl_4 \cdot 3L$	75–77	46.7 (45.4)	6.0 (5.5)	6.0 (5.9)	970m (10)		390w	–	325w	1600w
p-$FC_6H_4NMe_2O$	$Ph_3SnCl \cdot L$	81–83	56.3 (57.8)	4.3 (4.6)	2.4 (2.5)	280s,b (−5)		350w	270m	235w	1600w
	$Me_2SnCl_2 \cdot 2L$	85–87				990m (−15)		390w	555m	275w	1585w
	$Ph_2SnCl_2 \cdot L$	93–95	47.4 (48.0)	3.9 (4.0)	2.6 (2.8)	970m (5)		345w	280w	310w	1600w
m-$ClC_6H_4NMe_2O$	$Me_3SnCl \cdot L$	oil				955w (19)		350w	555w	285w	1600w,b
	$Ph_3SnCl \cdot L$	121–123	56.0 (56.0)	4.4 (4.5)	2.3 (2.5)	970m (4)		340w	285w	300w	1600m,b
	$SnCl_4 \cdot 3L$	oil				957s,sh (14)		390m,sh	–	305s,sh	1595w
p-$ClC_6H_4NMe_2O$	$Ph_3SnCl \cdot L$	80–82	55.4 (56.0)	4.2 (4.5)	2.3 (2.5)	990s, (−17),	950w –	335m	280m	240w	1585w
	$Me_2SnCl_2 \cdot 2L$	oil				985w, (−12),	952s –	360w	570s,sh	245w	1585w
	$Ph_2SnCl_2 \cdot L$	98–100	46.1 (46.5)	3.8 (3.8)	2.5 (2.7)	967w (6)		330w	285w	305m	1585w
	$SnCl_4 \cdot 3L$	78–80				980m (−7)		390m	–	325w	1585w
p-$BrC_6H_4NMe_2O$	$Ph_3SnCl \cdot L$	88–90	51.0 (51.8)	4.1 (4.1)	2.1 (2.3)	990s, (−17),	970w (3)	351w	280m	235w	1590m
	$Me_2SnCl_2 \cdot 2L$	95–97				990w (−17)		360w	555w	250w	1590m
	$Ph_2SnCl_2 \cdot L$	104–106	42.5 (42.8)	3.7 (3.5)	2.3 (2.5)	950w (23)		330w	285w	305s	1585w
	$SnCl_4 \cdot 3L$	76–78				962 (11)		360w	–	310w	1580m
p-$NO_2C_6H_4NMe_2O$	$Ph_3SnCl \cdot L$	89–91				952m (25)		335w	290w	245w	1595s
	$Me_2SnCl_2 \cdot 2L$	99–101	37.4 (37.0)	4.4 (4.45)	10.1 (9.6)	270m, (7)	955s –	370	570m	280m	1590s
	$Ph_2SnCl_2 \cdot L$	104–106	44.8 (45.6)	3.9 (3.8)	4.9 (5.3)	970m, (7)	950w, –	345m	290m	250m	1590w

Table 1 (continued)

Ligand (L)	Complex	m.p. (°C)	Analysis (found(calc.) (%))				IR data [a] (cm^{-1})			
			C	H	N	ν(N–O) (Δν)	ν(Sn–O)	ν(Sn–C)	ν(Sn–Cl)	ν(C=C)
m-MeOC$_6$H$_4$NMe$_2$O	Ph$_3$SnCl·L	oil				975w (−5)	330w	290m	250w	1595w
	SnCl$_4$·3L	oil				965w (5)	390w	–	330s,b	1595w
o-PhC$_6$H$_4$NMe$_2$O	Ph$_3$SnCl·L	83–84				975w (−5)	335w	280w	245w	1600w
	Ph$_2$SnCl$_2$·L	80–82				980w (−10)	330w	290w	240w	1590w
	SnCl$_4$·L	108–110	35.8 (35.5)	3.3 (3.1)	3.1 (3.0)					

[a] IR spectra recorded with Nujol mull; s, strong; m, medium; w, weak; sh, shoulder; b. broad.

Table 2

^{1}H NMR data; δ [a] (ppm) and $^2J(^{119}\text{Sn-CH})$ (Hz) for some tin complexes $R_xSnCl_{4-x}\cdot yL$

Ligand (L)	Complex	δ(N–Me)	δ(Ar)	δ(Me–Ar) (MeO)–	δ(Me–Sn)	$^2J(^{119}\text{Sn–CH})$
$C_6H_5NMe_2O$	$Me_3SnCl\cdot L$	3.55	7.2–7.9	–	0.65	66.0
	$Ph_3SnCl\cdot L$	3.40	7.1–7.85	–	–	–
	$Ph_2SnCl_2\cdot 2L$	3.65	7.0–7.89	–	–	–
	$PhSnCl_3\cdot 2L$	3.90	7.15–7.9	–	–	–
	$SnCl_4\cdot 3L$	4.0	7.0–8.1	–	–	–
o-$MeC_6H_4NHMe_2O$	$Me_3SnCl\cdot L$	3.85	7.1–7.9	2.65	0.65	64.0
	$Ph_3SnCl\cdot L$	3.65	7.0–8.0	2.65		
	$Ph_2SnCl_2\cdot L$	3.50	7.0–7.6	2.30		
	$PhSnCl_3\cdot 2L$	3.20	7.0–7.9	2.50		
	$SnCl_4\cdot 3L$	3.20	7.2–7.8	2.65		
m-$MeC_6H_4NMe_2O$	$Ph_3SnCl\cdot L$	3.74	7.0–7.75	2.85		
	$SnCl_4\cdot 3L$	3.75	7.1–7.9	2.84		
p-$MeC_6H_4NMe_2O$	$Me_3SnCl\cdot L$	3.60	7.05–7.8	2.43	0.70	65.0
	$Ph_3SnCl\cdot L$	3.35	7.0–7.9	2.35		
	$Me_2SnCl_2\cdot 2L$	3.60	7.1–7.8	2.40	1.05	118
	$Ph_2SnCl_2\cdot L$	3.65	7.0–7.85	2.30		
	$PhSnCl_3\cdot 2L$	3.80	7.1–8.2	2.35		
	$SnCl_4\cdot 3L$	3.90	7.05–7.85	2.40		
p-$FC_6H_4NMe_2O$	$Me_3SnCl\cdot L$	3.65	7.0–8.05		0.70	64.2
	$Ph_3SnCl\cdot L$	3.45	7.0–7.8			
	$Me_2SnCl_2\cdot 2L$	3.75	7.0–8.0		1.20	102
	$Ph_2SnCl_2\cdot L$	3.50	7.0–8.05			
m-$ClC_6H_4NMe_2O$	$Me_3SnCl\cdot L$	3.60	7.25–8.0		0.70	64
	$Ph_3SnCl\cdot L$	3.45	7.2–8.0			
p-$ClC_6H_4NMe_2O$	$Ph_3SnCl\cdot L$	3.45	7.0–7.9			
	$Me_2SnCl_2\cdot 2L$	3.80	7.1–8.0		1.20	116
	$Ph_2SnCl_2\cdot L$	3.60	7.1–7.7			
	$SnCl_4\cdot 3L$	3.80	7.1–8.1			
p-$BrC_6H_4NMe_2O$	$Ph_3SnCl\cdot L$	3.45	7.0–7.8			
	$Me_2SnCl_2\cdot 2L$	3.75	7.1–7.7		1.35	110
	$Ph_2SnCl_2\cdot L$	3.50	7.0–8.0			
p-$NO_2C_6H_4NMe_2O$	$Ph_3SnCl\cdot L$	3.70	7.1–7.8			
	$Ph_2SnCl_2\cdot L$	3.65	7.2–7.7			
m-$MeOC_6H_4NMe_2O$	$Ph_3SnCl\cdot L$	3.55	7.1–7.8	3.80		
	$SnCl_4\cdot 3L$	3.70	7.1–7.9	3.90		
o-$PhC_6H_4NMe_2O$	$Ph_3SnCl\cdot L$	3.40	7.1–7.6			
	$Ph_2SnCl_2\cdot L$	3.50	7.0–7.8			
	$SnCl_4\cdot L$	3.60	7.2–7.9			

[a] Downfield from internal TMS, in $CDCl_3$.

OMe, Ph) were prepared as illustrated in Scheme 1. The complexes were isolated as either white solids or oils. Their melting points (where relevant), elemental analyses, and IR spectral data are listed in Table 1, and their ^{1}H NMR spectral data in Table 2. The ^{119}Sn and ^{13}C NMR data for three representative complexes are listed in Table 3.

We have shown [3,4,7] that oxygen-donor ligands coordinate smoothly with tin(IV) compounds to give complexes of the type $R_xSnX_{4-x}\cdot L_n$. The number, n, of molecules of coordinated ligand can depend on the ratio of anionic ligands (X)

Table 3

^{119}Sn[a] and ^{13}C[b] NMR data; δ(ppm) and $^{1}J(^{119}\text{Sn}-^{13}\text{C})$ (Hz) for some tin complexes $R_xSnCl_{4-x}\cdot yL$, in $CDCl_3$.

Compound	δ^{119}Sn	^{13}C NMR									
		Tin compound				Ligand					
		(δC1) ^{1}J(Sn–C)	(δC2) ^{2}J(Sn–C)	(δC3) ^{3}J(Sn–C)	(δC4) ^{4}J(Sn–C)	(δC1)	(δC2)	(δC3)	(δC4)	δMe	Others
Ph_3SnCl (free)	−48.0[c]	(137.3)[d] 614.7	(136.2)[d] 50	(129.2)[d] 64.7	(130.5)[d] 11.8						
$Ph_3SnCl\cdot ON(Me_2)C_6H_4F$-*p*	−126.2	(140) –	(136.2) 48.3	(128.8) 65.4	(129.9) –	162.2	115.9	121.9	149.8	62.8	$^{1}J(^{19}\text{F}-\text{C}) = 250$ $^{2}J(^{19}\text{F}-\text{C}) = 23$ $^{3}J(^{19}\text{F}-\text{C}) = 9.0$
$Ph_3SnCl\cdot ON(Me_2)C_6H_4Cl$-*p*	−109.1	(140) –	(136.1) 47	128.8 64	129.9 –	152.7	121.4	135.1	129.2	62.7	
Ph_2SnCl_2(free)	−32.0[c]	(136.8)[d] 786.8	(134.9)[d] 63.3	(129.7)[d] 86.8	(131.8)[d] 17.7						
$Ph_2SnCl_2\cdot ON(Me_2)C_6H_4Cl$-*p*	[e]	(142.1) –	(135.4) 65	(129.9) 86	130.6 –	148.5	121.0	136.4	129.9	61.4	
$Ph_2SnCl_2\cdot ON(Me_2)C_6H_4NO_2$-*p*	−46.50	(142.5) –	(136.1) –	(129.1) –	(130.4) –	158.4	121.3	125.1	148.5	67.1	

[a] Downfield from external Me_4Sn. [b] Downfield from external TMS. [c] Data taken from ref. [10]. [d] Data taken from ref. [3]. [e] Not recorded.
For ^{13}C NMR data of the free ligand see ref. 16 and 17.

(X = halide or pseudohalide) to R groups in the tin compound. For example, triorganotin(IV) compounds form discrete penta-coordinate complexes, diorganotin (IV) compounds usually form hexa-coordinate complexes, and monoorganotin(IV) compounds usually form hexa-coordinate complexes. R_4Sn compounds do not form complexes with L.

We have now shown that, the ligands *N,N*-dimethylaniline *N*-oxides likewise coordinate with tin(IV) compounds via oxygen, to form complexes with tin in various coordination states depending on the nature of the tin(IV) compound. Thus the compounds R_3SnCl (R = Me, Ph) form penta-coordinate complexes and Me_2SnCl_2 and $PhSnCl_3$ hexa-coordinate complexes. The diorganotin halide Ph_2SnCl_2 forms penta-coordinate complexes with most of the ligands used, but, exceptionally with the parent oxide, $C_6H_5NMe_2O$, it forms a hexa-coordinate complex; behaviour that can be accounted for in terms of steric factors. The tetrahalide $SnCl_4$ is found to form hepta-coordinated complexes with all the ligands used except *o*-$PhC_6H_4NMe_2O$, which gives a penta-coordinate complex, $SnCl_4 \cdot L$; no doubt because of steric factors.

Infrared spectra

The IR data in Table 1 show that complexes are formed. There are changes in the stretching frequencies of Sn–C, Sn–Cl and C=C bonds upon complexation, but especially significant is the appeareance of a new band at ca. 350 cm^{-1} which serves as a good indicator of coordination. Moreover, the shift in the ν(NO) frequency is clear evidence for interaction between the NO group of the ligand and the tin compound. However coordination can shift the NO stretching frequency to either lower or higher field, and values of (ν(NO(ligand)) – ν(NO(complex)) show no systematic variations (Table 1).

Nuclear magnetic resonance

For the methyltin complexes, namely $Me_3SnCl \cdot L$ and $Me_2SnCl_2 \cdot 2L$, the ratio of the integrals of the signals from methyl protons of the ligand to those from the protons on the methyl groups on tin, provides a reliable measure of the number of coordinated ligands. Both the chemical shifts (δ ppm) and the ^{119}Sn–CH and ^{119}Sn–^{13}C coupling constants (J Hz) for any organotin compound were drastically affected on donor interaction, particularly the J values, and the changes can be used to estimate the coordination number, and so the number of coordinated ligands. It has been shown previously [3,8] that the J value can be used as an indicator of the coordination number, and e.g., the $^2J(^{119}$Sn–CH) values (ca. 65 Hz) observed for the Me_3SnCl-complexes in the present work (can be confidently assigned to the penta-coordinate complexes, i.e. $Me_3SnC \cdot L$. Similarly the $^2J(^{119}$Sn–CH) values (ca. 115 Hz) for Me_2SnCl_2 complexes clearly indicate hexa-coordination, i.e., $Me_2SnCl_2 \cdot 2L$. The larger the coupling constant, the higher the coordination number of tin.

The ^{13}C NMR spectral data for some tin complexes were recorded to provide an additional indicator for the coordination number in the case of complexes containing organic groups other than methyl, namely the phenyltin complexes (Table 3). The signal for the ipso carbon (the carbon atom attached to tin) of the phenyl group of $Ph_3SnCl \cdot L$ or $Ph_2SnCl_2 \cdot L$ was shifted downfield (by ca. 3 ppm), just enough to allow assignment as penta-coordinate complexes, whereas for hexa-coordinate $Ph_2SnCl_2 \cdot 2L$, the downfield shift is usually around 15 ppm. Unfortunately, the value of $^1J(^{119}Sn-^{13}C)$ could not be obtained because the poor signal to noise ratio prevented observation of the tin satellites. The ^2J and $^3J(^{119}Sn-^{13}C)$ values were not significantly affected.

The ^{119}Sn NMR chemical shift is a very sensitive to complexation, and is usually greatly shifted downfield or upfield on bonding to a Lewis base [9]. We thus recorded the ^{119}Sn NMR spectra for some phenyltin complexes (Table 3) in order to obtain additional support for our conclusions. The ^{119}Sn chemical shift for Ph_3SnCl (-48 ppm) [10] was shifted upfield to -109 ppm on complexation with *p*-$ClC_6H_4NMe_2O$ and to -126 ppm on complexation with *p*-$FC_6H_4NMe_2O$; the difference (17 ppm) between the δ ^{119}Sn shifts for the complexes can be attributed to the differences in the electronic effect of the two halogens. The compound Ph_2SnCl_2 (δ $^{119}Sn = 32$ ppm) [10] seems to give a complex with *p*-$NO_2C_6H_4NMe_2O$, and the value of δ ^{119}Sn (-46.5 ppm) for the this complex is consistent with penta-coordination at Sn, i.e. with a $Ph_2SnCl_2 \cdot L$ species.

No stable complexes could be obtained from the compounds $^nBu_3SnCl$ and $^nBu_2SnCl_2$ with the ligands used in this work, probably for steric reasons.

Experimental

General

^1H NMR spectra were recorded at 25 ° C on a Hitachi Perkin Elmer R-248 high resolution NMR spectrometer 60 MHz, with the deuterium signal of the solvent as lock and reference.

Natural abundance, proton-decoupled FT ^{13}C and ^{119}Sn NMR spectra were recorded at room temperature on a Bruker 300 MHz spectrometer at Dortmund University, Dortmund, FRG.

IR spectra were recorded on an SP 2000 spectrometer over the range 200–4000 cm^{-1} with Nujol mulls and CsI discs.

Analyses of the complexes were carried out with a CHN analyser, type 1106 (Carlo Erba) at Dortmund University.

Preparation of compounds

Starting materials

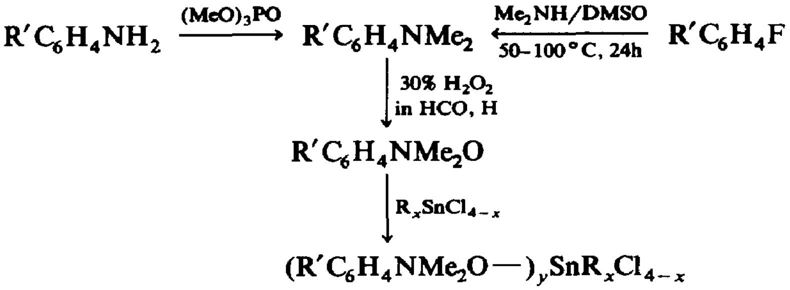
The compounds Me_4Sn, Ph_4Sn, $SnCl_4$ and Ph_3SnCl were commercial products. The compounds R_xSnCl_{4-x} ($R = Me$, $x = 2, 3$; $R = Ph$, $x = 1, 2$) were prepared by standard methods [11,12].

N,N-Dimethylaniline oxides R'-$C_6H_4NMe_2O$, were prepared as shown in Scheme 1; the *N,N*-dimethylaniline was made by methylation [13,14] and conversion into the *N,N*-dimethylaniline *N*-oxide (L) by treatment with H_2O_2 [15].

The identities of the organotin (IV) compounds, the tertiary amines, and their oxides were confirmed comparison of their physical propertiers with those in the literature [15].

Preparation of $R_xSnCl_{4-x} \cdot y(O(Me_2)\ NC_6H_4R')$ *complexes.* These were made by the following general method.

The tin compound R_xSnCl_{4-x} (1 mmol) was dissolved in chloroform (5 ml) and a solution of the oxide ($R'C_6H_4NMe_2O$) (in slight excess) in chloroform (5 ml) was added in one portion at 0 ° C and the mixture shaken for ca. 5 min. The solution



Scheme 1. Preparation of *N,N*-dimethylaniline *N*-oxide complexes of tin (IV) compounds. For R', R, *x*, *y* see text.

was allowed to evaporate slowly at room temperature until the volume was reduced to 5 ml. Light petroleum (b.p. 60–80 °C) was added to the point of turbidity, and the mixture stirred for ca. 3 h until the product had completely separated out. When the product was solid, it was filtered off, washed with light petroleum, and dried under vacuum for several hours. When it was an oil, it was separated from the mother liquour by decantation, washed with light petroleum, and dried. The oil sometimes solidified when stirred with light petroleum overnight, and then could be treated as for the solids above, but when it remained as an oil, it was pumped hard for several hours, to give a product pure enough for investigation.

Acknowledgements

The authors are grateful to Prof. Dr. W.P. Neumann, Organische Chemie 1, Dortmund Universität, 4600 Dortmund 50, FRG for assistance in recording of the ^{13}C and ^{119}Sn NMR spectra and for arranging the CHN analyses. The University of Mosul, College of Science is thanked for awarding M.A.M. Al-Tayy a grant to enable him to work for the M.Sc. degree.

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