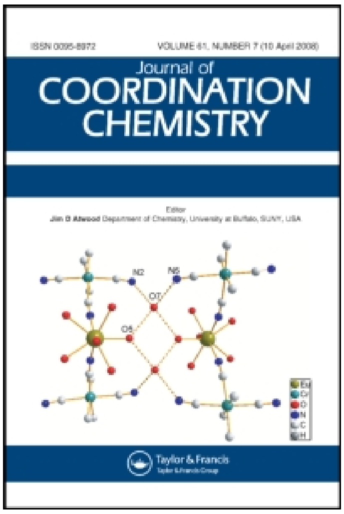

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# SYNTHESIS AND CHARACTERIZATION OF OUTER COORDINATION SPHERE COMPLEXES: [LH ]$_2$[SnC$^{2-}_6$], [LH ]$_2$[Me$_4$Sn$_2$Cl$^{2-}_6$] AND [LH ][Ph$_2$SnCl$_3^-$](LH = PROTONATED 2-METHOXY-1-[(4-CHLORO-PHENYLIMINO)METHYL]NAPHTHALENE). CRYSTAL STRUCTURE OF 2-METHOXY-1-[(4-CHLOROPHENYLIMINO)-METHYLJNAPHTHALENIUM TRICHLORODIPHENYLSTANNATE(IV), [LH ][Ph$_2$SnCI$_3^-$], DERIVED FROM DIPHENYLTIN DICHLORIDE AND 2-METHOXY-l-[(4-CHLOROPHENYLIMINO)-METHYL]NAPHTHALENE

Guan-yeow Yeap[a]; Ai-chen Ng[a]; Siang-guan Teoh[a]; Soon-beng Teo[a]; Jean-paul Declercq[b]
[a] School of Chemical Sciences, Universiti Sains Malaysia, Penang, Malaysia [b] Laboratoire de Chimie Physique et de Cristallographie, Universite Catholique de Louvain, Louvain-la-Neuve, Belgium

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# SYNTHESIS AND CHARACTERIZATION OF OUTER COORDINATION SPHERE COMPLEXES: $[LH^+]_2[SnCl_6^{2-}]$, $[LH^+]_2[Me_4Sn_2Cl_6^{2-}]$ AND $[LH^+][Ph_2SnCl_3^-]$($LH^+$ = PROTONATED 2-METHOXY-1-[(4-CHLOROPHENYLIMINO)METHYL]NAPHTHALENE). CRYSTAL STRUCTURE OF 2-METHOXY-1-[(4-CHLOROPHENYLIMINO)-METHYL]NAPHTHALENIUM TRICHLORODIPHENYLSTANNATE(IV), $[LH^+][Ph_2SnCl_3^-]$, DERIVED FROM DIPHENYLTIN DICHLORIDE AND 2-METHOXY-1-[(4-CHLOROPHENYLIMINO)-METHYL]NAPHTHALENE

GUAN-YEOW YEAP[a,*], AI-CHEN NG[a], SIANG-GUAN TEOH[a], SOON-BENG TEO[a] and JEAN-PAUL DECLERCQ[b]

[a]*School of Chemical Sciences, Universiti Sains Malaysia, 11800 Penang, Malaysia;* [b]*Laboratoire de Chimie Physique et de Cristallographie, Universite Catholique de Louvain, Place L. Pasteur 1, B-1348 Louvain-la-Neuve, Belgium*

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The reactions of $SnCl_4$, $Me_2SnCl_2$ and $Ph_2SnCl_2$ with the Schiff base (L) 2-methoxy-1-[(4-chlorophenylimino)methyl]naphthalene result in the formation of tin(IV) anionic complexes. $Ph_2SnCl_2$ reacts with the Schiff base in dichloromethane to form the complex salt

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[*] Corresponding author.

$[LH^+][Ph_2SnCl_3^-]$ ($LH^+$ = protonated L). X-ray structural analysis has been carried out on a single crystal of $[LH^+][Ph_2SnCl_3^-]$. The reactions of $SnCl_4$ and $Me_2SnCl_2$ with the Schiff base form the complexes $[LH^+]_2[SnCl_6^{2-}]$ and $[LH^+]_2[Me_4Sn_2Cl_6^{2-}]$, respectively. All complex salts were characterized by elemental analysis and spectroscopic methods (IR and $^1H$ NMR).

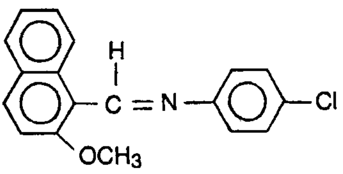
*Keywords:* Diphenyltin(IV); Crystal structure; Schiff base

## INTRODUCTION

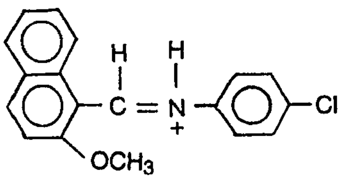
Reactions of inorganic tin(IV) and organotin(IV) chlorides and isothiocyanate with aromatic Schiff bases containing various substituents in the aniline as well as aldehyde fragments have been studied extensively over the last two and a half decades.[1–11] Earlier investigation has established that the reactions which involve Schiff bases salicylideneaniline and 2-hydroxy-1-naphthalideneaniline usually lead to the formation of adducts which exhibit Sn–O and Sn–N coordination modes. However, recent studies show that reactions of tin compounds with Schiff bases in which the 2-OH in the aldehyde fragment is removed or substituted by 4-OH, 2-$OCH_3$ and 4-$OCH_3$ result in the formation of complex salts.[12–14] Interaction of $Ph_2SnCl_2$ with Schiff bases without the 2-OH substituent led to the formation of the complex salt $[LH^+]_2[Ph_2SnCl_4^{2-}]$ in which L is *N*-(4-hydroxybenzalidene)-4-methoxyaniline.[12]

In this paper, we report the reactions of $SnCl_4$, $Me_2SnCl_2$ and $Ph_2SnCl_2$ with 2-methoxy-1-[(4-chlorophenylimino)methyl]naphthalene wherein the aniline fragment contains a lateral substituent, 4-Cl. An X-ray structural determination has performed on a single crystal of 2-methoxy-1-[4-chlorophenylimino)methyl]naphthalenium trichlorodiphenylstannate(IV). All synthesized complex salts were examined by microanalysis, infra-red and $^1H$ NMR spectroscopic methods.

The ligand used and the protonated Schiff base thus obtained are as follows.



[2MeONAP-4Cl]



[2MeONAPH-4Cl+]

## EXPERIMENTAL

### Reagents

2-Methoxy-1-naphthaldehyde was obtained from Fluka Chemie AG; 4-chloroaniline, inorganic tin(IV) and organotin(IV) chlorides were supplied by Aldrich Chemical Company.

### Preparation of Ligands

2-Methoxy-1-[(4-chlorophenylimino)methyl]naphthalene was prepared by adding equimolar amounts of 4-chloroaniline to 2-methoxy-1-naphthaldehyde in 95% ethanol.

### Preparation of Complexes

All the complex salts were readily prepared by the reactions of the tin compounds with 2-methoxy-1-[(4-chlorophenylimino)methyl]naphthalene. The preparation of 2-methoxy-1-[(4-chlorophenylimino)methyl]naphthalenium trichlorodiphenylstannate(IV) is described as an example.

A solution of 2-methoxy-1-[(4-chlorophenylimino)methyl]naphthalene (1.7746 g, 6 mmol) in the 1 : 1 dichloromethane and methanol was added to diphenyltin dichloride (1.0314 g, 3 mmol) in the same solvent. The mixture was left standing at room temperature overnight whereupon yellowish crystals were precipitated. The compound thus obtained was recrystallized from dichloromethane and *n*-hexane.

### Measurements

All microanalysis were carried out at the School of Chemical Science, Universiti Sains Malaysia. Results are shown in Table I. The IR spectra of the Schiff base and complexes (Table V) were recorded on a Perkin Elmer

TABLE I Microanalytical data for 2MeONAP–4Cl and its complexes

| *Compound* | *Formula* | *Calculated* (%) | | | | *Found* (%) | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | *Sn* | *C* | *H* | *N* | *Sn* | *C* | *H* | *N* |
| 2MeONAP–4Cl | $C_{18}H_{14}NOCl$ | | 73.1 | 4.8 | 4.7 | | 73.1 | 4.8 | 4.7 |
| $[2MeONAP–4Cl^+][Ph_2SnCl_3^-]$ | $C_{30}H_{25}NOCl_4Sn$ | 17.6 | 53.3 | 3.7 | 2.1 | 17.6 | 53.3 | 3.7 | 2.1 |
| $[2MeONAP–4Cl^+]_2[Me_4Sn_2Cl_6^{2-}]$ | $C_{20}H_{21}NOCl_4Sn$ | 21.5 | 43.5 | 3.8 | 2.5 | 21.5 | 43.5 | 3.8 | 2.5 |
| $[2MeONAP–4Cl^+]_2[SnCl_6^{2-}]$ | $C_{36}H_{30}N_2O_2Cl_8Sn$ | 12.8 | 46.8 | 3.3 | 3.0 | 12.8 | 46.7 | 3.2 | 3.0 |

System 2000 FTIR spectrophotometer in the frequency range 4000–200 cm$^{-1}$ with samples in KBr discs. $^{1}$H NMR data were collected using a Bruker AC-P 300 MHz NMR spectrometer with deuterated DMSO-$d_6$ as the solvent and tetramethylsilane as internal standard. Selected $^{1}$H NMR peaks are presented in Table VI.

### Crystallography: Determination of the X-ray Crystal Structure of 2-Methoxy-1-[(4-chlorophenylimino)methyl]naphthalenium Trichlorodiphenylstannate(IV)

Intensity data for a crystal with dimensions 0.42 × 0.38 × 0.19 mm were measured at room temperature on a Huber diffractometer fitted with graphite monochromated MoK$\alpha$ radiation, $\lambda = 0.71069$ Å. The $\omega$ scan was employed to measure data to $\theta_{max} \leq 27.5°$. No significant decomposition occurred during the data collection. Corrections were applied for Lorentz and polarization effects but not for absorption. Data which satisfied the $F > 2.5\sigma(F)$ criterion of observability were used in the subsequent analysis. Crystal data are listed in Table II.

The structure was solved using SHELXS86[15] and refined by a full-matrix least-squares procedure based on $F$ using SHELXS76.[16] 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

TABLE II Crystal data and structure refinement details for $[2MeONAPH\text{-}4Cl^{+}][Ph_2SnCl_3^{-}]$

| | |
|---|---|
| Empirical formula | $C_{30}H_{25}C_{14}NOSn$ |
| Formula weight | 676.00 |
| Crystal system | Monoclinic |
| Space group | $P2_1/n$ |
| $a$ | 7.8050(11) Å |
| $b$ | 18.066(3) Å |
| $c$ | 20.737(3) Å |
| $\beta$ | 93.860(13) |
| $V$ | 2917.4(7) Å$^3$ |
| $Z$ | 4 |
| $D_c$ | 1.539 mg m$^{-3}$ |
| $F(000)$ | 1352 |
| $\theta$ range for data collection | 2.25–27.51° |
| Data collected | 6693 |
| $R$ | 0.0567 |
| $R_w$ | 0.0938 |
| $w$ | $k/[\sigma^2(F_0) + gF_0^2]$ |
| $k$ | 1 |
| $g$ | 0.0012 |

isotropically. Final atomic coordinates are given in Table III. Analysis of variance showed no special features and the maximum residual electron density peak in the final difference map was 0.72 eÅ$^3$ (near the tin atom). The scattering factors for all the atoms used were as incorporated in SHELXS76. Interatomic bond distance and angles are listed in Table IV. Lists of hydrogen atomic coordinates, thermal parameters and observed and calculated structure factors are available on request from the authors.

TABLE III Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (Å$^2 \times 10^3$) for [2MeONAPH-4Cl$^+$][$Ph_2SnCl_3^-$]. $U(eq)$ is defined as one-third of the trace of the orthogonalized $U_{ij}$ tensor

| | *x/a* | *y/b* | *z/c* | *U(eq)* |
|---|---|---|---|---|
| Sn | 477(1) | 1861(1) | 2041(1) | 43(1) |
| Cl(1) | 2188(2) | 2456(1) | 1175(1) | 59(1) |
| Cl(2) | −1046(2) | 1230(1) | 2945(1) | 63(1) |
| Cl(3) | 2892(2) | 1101(1) | 2296(1) | 68(1) |
| Cl(4) | 7022(3) | 3418(1) | 4336(1) | 18(1) |
| C(5) | −1244(7) | 1389(3) | 1315(2) | 44(1) |
| C(6) | −2990(7) | 1318(3) | 1406(3) | 57(1) |
| C(7) | −4096(8) | 1037(4) | 923(3) | 71(2) |
| C(8) | −3509(10) | 805(4) | 355(3) | 77(2) |
| C(9) | −1788(9) | 861(4) | 253(3) | 72(2) |
| C(10) | −672(8) | 1164(3) | 738(3) | 59(2) |
| C(11) | 530(7) | 2870(3) | 2563(3) | 53(1) |
| C(12) | 1038(9) | 2872(4) | 3219(3) | 71(2) |
| C(13) | 1146(12) | 3539(6) | 3549(4) | 108(3) |
| C(14) | 728(13) | 4184(5) | 3242(6) | 119(4) |
| C(15) | 202(13) | 4180(4) | 2610(5) | 110(3) |
| C(16) | 121(10) | 3536(4) | 2268(4) | 76(2) |
| N(17) | 4397(6) | 3954(3) | 1651(2) | 60(1) |
| C(18) | 3805(7) | 4559(4) | 1388(3) | 56(2) |
| C(19) | 3247(9) | 4670(4) | 721(3) | 69(2) |
| C(20) | 3674(11) | 4164(5) | 251(3) | 86(3) |
| C(21) | 3166(14) | 4312(7) | −418(4) | 120(5) |
| C(22) | 2327(14) | 4940(8) | −572(4) | 135(6) |
| C(23) | 1876(10) | 5479(6) | −129(3) | 98(3) |
| C(24) | 964(13) | 6138(8) | −303(4) | 127(5) |
| C(25) | 560(12) | 6624(7) | 127(5) | 122(4) |
| C(26) | 978(10) | 6511(6) | 774(4) | 100(3) |
| C(27) | 1838(8) | 5871(4) | 981(3) | 73(2) |
| C(28) | 2334(8) | 5343(5) | 542(3) | 73(2) |
| C(29) | 5027(7) | 3847(3) | 2309(3) | 54(1) |
| C(30) | 5198(9) | 4425(4) | 2737(3) | 72(2) |
| C(31) | 5837(9) | 4281(4) | 3363(3) | 75(2) |
| C(32) | 6262(9) | 3577(4) | 3545(3) | 71(2) |
| C(33) | 6115(10) | 3005(4) | 3113(4) | 79(2) |
| C(34) | 5462(9) | 3143(4) | 2490(4) | 70(2) |
| O(35) | 4542(8) | 3566(3) | 446(2) | 102(2) |
| C(36) | 4907(14) | 2996(5) | −16(5) | 144(5) |

TABLE IV Selected bond lengths (Å) and angles (°) for [2MeONAPH–4Cl$^+$][Ph$_2$SnCl$_3^-$]

| | | | |
|---|---|---|---|
| Sn–C(11) | 2.118(5) | C(18)–C(19) | 1.437(8) |
| Sn–C(5) | 2.127(5) | C(19)–C(20) | 1.392(10) |
| Sn–Cl(3) | 2.363(2) | C(19)–C(28) | 1.445(10) |
| Sn–Cl(1) | 2.5472(13) | C(20)–O(35) | 1.322(11) |
| Sn–Cl(2) | 2.5544(14) | C(20)–C(21) | 1.442(11) |
| Cl(4)–C(32) | 1.731(7) | C(21)–C(22) | 1.34(2) |
| C(5)–C(10) | 1.368(7) | C(22)–C(23) | 1.40(2) |
| C(5)–C(6) | 1.394(8) | C(23)–C(24) | 1.421(14) |
| C(6)–C(7) | 1.375(8) | C(23)–C(28) | 1.435(9) |
| C(7)–C(8) | 1.360(10) | C(24)–C(25) | 1.31(2) |
| C(8)–C(9) | 1.377(9) | C(25)–C(26) | 1.376(11) |
| C(9)–C(10) | 1.397(8) | C(26)–C(27) | 1.391(10) |
| C(11)–C(16) | 1.378(9) | C(27)–C(28) | 1.392(10) |
| C(11)–C(12) | 1.392(8) | C(29)–C(34) | 1.363(8) |
| C(12)–C(13) | 1.385(11) | C(29)–C(30) | 1.370(8) |
| C(13)–C(14) | 1.357(13) | C(30)–C(31) | 1.385(8) |
| C(14)–C(15) | 1.346(13) | C(31)–C(32) | 1.361(9) |
| C(15)–C(16) | 1.361(10) | C(32)–C(33) | 1.367(10) |
| N(17)–C(18) | 1.293(7) | C(33)–C(34) | 1.380(10) |
| N(17)–C(29) | 1.433(7) | O(35)–C(36) | 1.449(8) |
| C(11)–Sn–C(5) | 134.1(2) | C(20)–C(19)–C(18) | 120.7(8) |
| C(11)–Sn–Cl(3) | 113.4(2) | C(20)–C(19)–C(28) | 120.7(7) |
| C(5)–Sn–Cl(3) | 112.4(2) | C(18)–C(19)–C(28) | 118.4(6) |
| C(11)–Sn–Cl(1) | 90.2(2) | O(35)–C(20)–C(19) | 117.6(7) |
| C(5)–Sn–Cl(1) | 90.37(13) | O(35)–C(20)–C(21) | 123.2(8) |
| Cl(3)–Sn–Cl(1) | 87.39(6) | C(19)–C(20)–C(21) | 119.1(9) |
| C(11)–Sn–Cl(2) | 90.2(2) | C(22)–C(21)–C(20) | 119.1(10) |
| C(5)–Sn–Cl(2) | 92.14(14) | C(21)–C(22)–C(23) | 125.1(10) |
| Cl(3)–Sn–Cl(2) | 88.85(6) | C(22)–C(23)–C(24) | 124.1(9) |
| Cl(1)–Sn–Cl(2) | 176.07(5) | C(22)–C(23)–C(28) | 117.3(10) |
| C(10)–C(5)–C(6) | 118.1(5) | C(24)–C(23)–C(28) | 118.5(9) |
| C(10)–C(5)–Sn | 120.7(4) | C(25)–C(24)–C(23) | 122.0(9) |
| C(6)–C(5)–Sn | 121.1(4) | C(24)–C(25)–C(26) | 121.0(11) |
| C(7)–C(6)–C(5) | 120.4(6) | C(25)–C(26)–C(27) | 120.1(10) |
| C(27)–C(28)–C(23) | 117.0(8) | C(26)–C(27)–C(28) | 121.3(7) |
| C(8)–C(7)–C(6) | 120.9(6) | C(27)–C(28)–C(19) | 124.3(6) |
| C(7)–C(8)–C(9) | 120.0(6) | C(23)–C(28)–C(19) | 118.7(8) |
| C(8)–C(9)–C(10) | 119.1(6) | C(34)–C(29)–C(30) | 121.4(6) |
| C(5)–C(10)–C(9) | 121.5(6) | C(34)–C(29)–N(17) | 116.9(6) |
| C(16)–C(11)–C(12) | 118.2(6) | C(30)–C(29)–N(17) | 121.8(5) |
| C(16)–C(11)–Sn | 122.0(5) | C(29)–C(30)–C(31) | 118.5(6) |
| C(12)–C(11)–Sn | 119.8(5) | C(32)–C(31)–C(30) | 120.1(6) |
| C(13)–C(12)–C(11) | 119.3(8) | C(31)–C(32)–C(33) | 121.1(6) |
| C(14)–C(13)–C(12) | 120.6(8) | C(31)–C(32)–Cl(4) | 118.6(6) |
| C(15)–C(14)–C(13) | 120.0(8) | C(33)–C(32)–Cl(4) | 120.2(6) |
| C(14)–C(15)–C(16) | 120.7(9) | C(32)–C(33)–C(34) | 119.1(6) |
| C(15)–C(16)–C(11) | 121.0(8) | C(29)–C(34)–C(33) | 119.8(6) |
| C(18)–N(17)–C(29) | 127.3(5) | C(20)–O(35)–C(36) | 119.8(8) |
| N(17)–C(18)–C(19) | 126.8(6) | | |

## RESULTS AND DISCUSSION

The complex salts $[\mathrm{2MeONAPH{-}4Cl^+}]_2[\mathrm{SnCl_6^{2-}}]$, $[\mathrm{2MeONAPH{-}4Cl^+}]_2[\mathrm{Me_4Sn_2Cl_6^{2-}}]$ and $[\mathrm{2MeONAPH{-}4Cl^+}][\mathrm{Ph_2SnCl_3^-}]$ are thermally stable at ambient temperature.

The molecular structure of 2-methoxy-1-[(4-chlorophenylimino)methyl]-naphthalenium trichlorodiphenylstannate(IV) with numbering scheme is depicted in Figure 1. It is clear that the trichlorodiphenylstannate(IV) anion exhibits trigonal bipyramidal geometry with a Cl atom and the *ipso*-carbon atoms of the two phenyl groups lying in the equatorial plane. The remaining two Cl atoms occupy apical sites. The average axial Sn–Cl bond distance (2.551(1) Å) is shorter than that in the stannate anions $[\mathrm{Ph_2SnCl_4^{2-}}]$ (2.603(1) Å) of *bis*[*N*-(4-hydroxybenzalidene)-4-methoxyanilinium] tetrachlorodiphenylstannate(IV).[12] This observation can be ascribed to the electron density around Sn in $\mathrm{Ph_2SnCl_3^-}$ which is less than that in

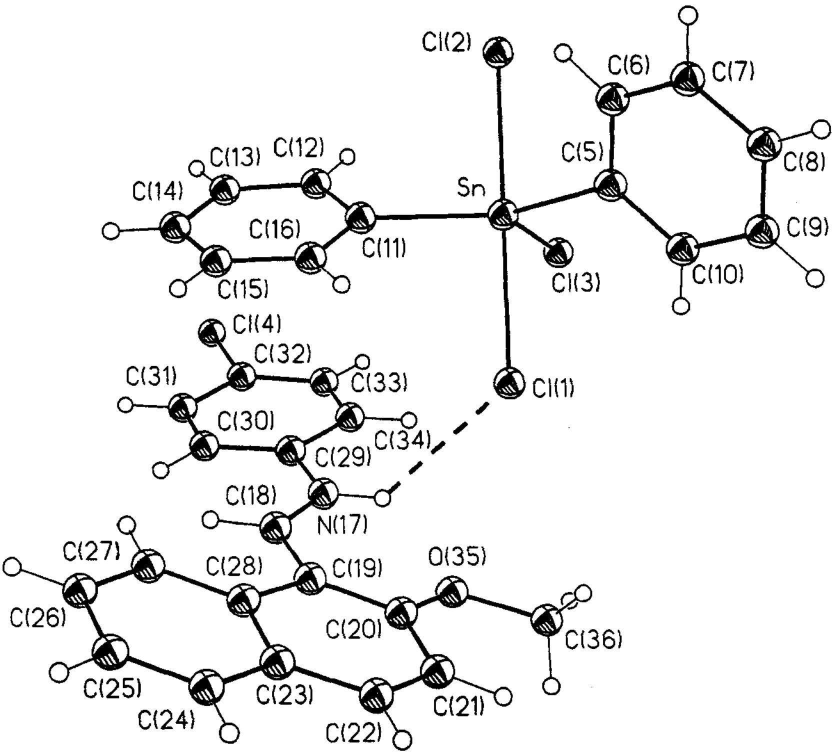


FIGURE 1 Molecular structure with atom numbering scheme for $[\mathrm{2MeONAPH{-}4Cl^+}][\mathrm{Ph_2SnCl_3^-}]$.

$[Ph_2SnCl_4^{2-}]$. As a result, electronic repulsion between the Sn and Cl atoms is significantly lower, leading to the shortening of the Sn–Cl bond length in $Ph_2SnCl_3^-$, as exemplified by *d*-orbital population analysis.[17,18]

The uninegatively charged $Ph_2SnCl_3^-$ species is found to be counteracted by a unipositively charged Schiff base. This is evident by the H(N17)···Cl(1) distance (2.657 Å) indicating the presence of hydrogen bonding between $Ph_2SnCl_3^-$ and the protonated Schiff base.[19,20] The charge on the Schiff base is a result of protonation of the imine nitrogen atom as known to occur in $[S_2N_2H^+]$[21] and *bis*[*N*-(4-hydroxybenzalidene)-4-methoxyanilinium] tetrachlorodiphenylstannate.[12] The presence of the protonated imine nitrogen is also substantiated by the IR data (Table V) wherein a significant shift of $\nu_{C=N}$ from 1620 to 1655 cm$^{-1}$ is observed. A new band at 3200 cm$^{-1}$ in the complex is attributable to the N–H stretching vibration. A peak in the $^1$H NMR spectrum which is assignable to the azomethine proton in the free ligand at $\delta = 9.42$ ppm is shifted upfield to 9.14 ppm upon complex formation. A new peak at $\delta = 10.69$ ppm which is not present in the ligand may be ascribed to the =N–H proton. Actually, this imine hydrogen has been located by a difference Fourier map and refined using isotropic displacement parameters to give a H···(N1) distance 0.86 Å.

For the complex prepared by the reaction of $Me_2SnCl_2$ with 2-methoxy-1-[(4-chlorophenylimino)methyl]naphthalene in dichloromethane, its microanalytical data is in accordance with the formulation $[2MeONAPH-4Cl^+]_2[Me_4SnCl_6^{2-}]$. A band which appears at 3160 cm$^{-1}$ in its IR spectrum (Table V) can be ascribed to the stretch of the N–H bond while its $^1$H NMR spectrum (Table VI) shows that the peak assignable to the azomethine

TABLE V Selected IR data (cm$^{-1}$) for 2MeONAP–4Cl and its complexes

| *Compound* | $\nu_{N-H}$ | $\nu_{C-N}$ | $\nu_{C-O}$ | |
|---|---|---|---|---|
| 2MeONAP–4Cl | | 1620 | 1245 | |
| $[2MeONAP-4Cl^+][Ph_2SnCl_3^-]$ | 3200 | 1655 | 1260 | 1175 |
| $[2MeONAP-4Cl^+]_2[Me_4Sn_2Cl_6^{2-}]$ | 3160 | 1650 | 1255 | 1165 |
| $[2MeONAP-4Cl^+]_2[SnCl_6^{2-}]$ | 3200 | 1660 | 1170 | |

TABLE VI Selected $^1$H chemical shift data (ppm) for 2MeONAP–4Cl and its complexes

| *Compound* | $\delta_{C=NH}$ | $\delta_{CH=N}$ |
|---|---|---|
| 2MeONAP–4Cl | | 9.42 |
| $[2MeONAP-4Cl^+][Ph_2SnCl_3^-]$ | 10.69 | 9.14 |
| $[2MeONAP-4Cl^+]_2[Me_4Sn_2Cl_6^{2-}]$ | 10.68 | 9.15 |
| $[2MeONAP-4Cl^+]_2[SnCl_6^{2-}]$ | 10.68 | 9.17 |

proton in the ligand has shifted upfield upon complexation, thus indicating a change in the hybridization of the nitrogen atomic orbital from $sp^2$ to $sp^3$. This evidence is further supported by the appearance of a new N–H proton peak at 10.68 ppm.

As we have reported in an earlier paper, in the complexes *bis*[*N*-(2-methoxy-1-naphthalidene)-3-methoxyanilinium] hexachlorotetramethyldistannate(IV) dihydrate,[14] the $Me_2SnCl_3^-$ anion crystallizes as a loosely associated dimer and not as a monomeric five-coordinate species. The present structure may be dimeric.

Microanalytical data for the complex derived from $SnCl_4$ and 2MeONAP–4Cl (Table I) are consistent with the formulation $[2MeONAPH{-}4Cl^+]_2[SnCl_6^{2-}]$. The similarities of the IR and $^1H$ NMR data for this complex with those of *bis*[*N*-benzalideneanilinium]hexachlorostannate(IV)[13] suggest the existence of two Schiff bases in the protonated form which are countered by the hexachlorostannate(IV) dianion formed from $SnCl_4$ and the chloride ions present in the dichloromethane solvent.

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