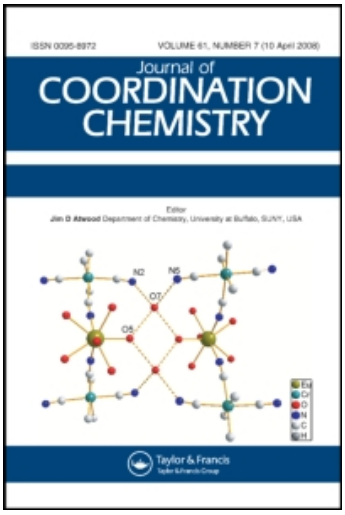

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Synthesis and spectroscopic studies of new organotin(IV) complexes with tridentate N- and O-donor Schiff bases

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The Schiff bases H_2L^1 and H_2L^2 have been prepared by the reaction of 2-amino-4-chlorophenol with pyrrole-2-carbaldehyde and 2-hydroxy-1-naphtaldehyde, respectively, and HL^3 from reaction of 2-(aminomethyl)pyridine with 2-hydroxy-1-naphtaldehyde. Organotin complexes $[SnPh_2(L^1)]$ (**1**), $[SnPh_2(L^2)]$ (**2**), $[SnMe_2(L^2)]$ (**3**) and $[SnPhCl_2(L^3)]$ (**4**) were synthesized from reaction of $SnPh_2Cl_2$ and $SnMe_2Cl_2$ with these Schiff bases. The synthesized complexes have been investigated by elemental analysis and FT-IR, ^{1}H NMR and ^{119}Sn NMR spectroscopy. In complexes the Schiff bases are completely deprotonated and coordinated to tin as tridentate ligands via phenolic oxygen, pyrrolic, and imine nitrogens in **1**, two phenolic oxygens and imine nitrogen in **2** and **3**, and phenolic oxygen, imine and pyridine nitrogens in **4**. The coordination number of tin in **1**, **2**, and **3** is five and in **4** is six.

Keywords: Organotin; Schiff base; ^{119}Sn NMR; N-donor ligands; O-donor ligands

1. Introduction

Metal complexes of Schiff bases show biological activities including antibacterial, antifungal, anticancer, and herbicidal. Schiff-base complexes have also been used in catalytic reactions and as biological models for understanding the structure of biomolecules [1–3]. Increasing attention has been devoted to Schiff-base complexes of organotin(IV) moieties in view of their potential applications in medicinal chemistry and biotechnology and their structural variety. Organotin(IV) complexes have been the subject of interest for some time because of their biomedical and commercial applications [4, 5], including *in vitro* and *in vivo* antitumor activity. Many organotin(IV) complexes have been found to be as effective as or even better than traditional anticancer drugs [5–9]. Recent studies showed that low doses of organotin compounds can exhibit antitumor activity and have suggested an action mode via gene-mediated pathway in the cancer cells, opening a new research sub-area on organotin compounds [10]. An interesting development in bioorganotin chemistry is introducing ligands which are themselves bioactive [11]. Schiff bases are potential anticancer drugs and some drugs have higher activity when administered as metal complexes than as free ligands.

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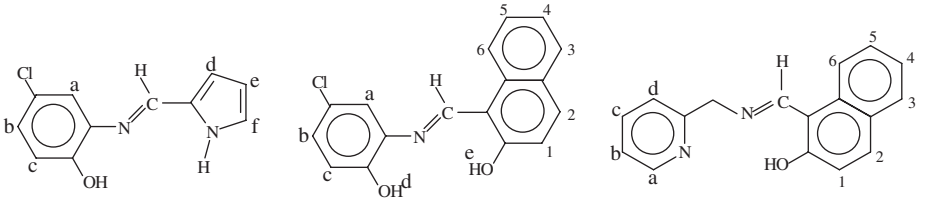


H_2L^1 H_2L^2 HL^3

Figure 1. Structure of Schiff bases.

Much work has been performed on synthesis and characterization of organotin(IV) complexes with Schiff bases due to their special pharmacological and antitumor activity [12–15]. In addition to antitumor activity, organotin(IV) complexes with Schiff bases presents an interesting variety of structural possibilities caused by the multidenticity of these ligands. Both aliphatic and aromatic Schiff bases in their neutral and deprotonated forms have been reacted with organotin(IV) halides; the complexes formed exhibit variable stoichiometry in the metal to ligand ratio and different modes of coordination [16–23].

In continuation of our studies on synthesis and characterization of organotin(IV) complexes [4, 17–19, 24, 25], we report the results of our investigations on ligating properties of three potentially multidentate Schiff-base ligands, 2-{[N-(5-chloro-2-hydroxyphenyl)imino]methyl}pyrrole (H_2L^1), 1-[N-(5-chloro-2-hydroxyphenyl)imino]-methyl}-2-naphthol (H_2L^2) and 1-[N-(2-pyridinylmethyl)imino]methyl}-2-naphthol (HL^3), figure 1, with diorganotin(IV) dichlorides, SnR_2Cl_2 (R = Me and Ph).

2. Experimental

Pyrrole-2-carbaldehyde, 2-amino-4-chlorophenol, 2-hydroxy-1-naphtaldehyde, 2-(amino-methyl)pyridine, triethylamine and dimethyltindichloride were purchased from Merck Company and diphenyltindichloride was purchased from Acros Company. All materials were used as received. All solvents were of reagent grade and used without purification. IR spectra were obtained using a FT BOMEM MB102 spectrophotometer. ^{1}H NMR spectra were recorded with a Bruker Avance DPZ500 spectrometer at 500.130 MHz using TMS as reference and $CDCl_3$ or DMSO-d_6 as solvent (conc. ~0.1 mM) at r.t. and spectral width 20 ppm. ^{119}Sn{^{1}H} NMR spectra were recorded with a Bruker Avance DPZ400 spectrometer at 149.211 MHz using $SnMe_4$ as reference and $CDCl_3$ as solvent (conc. ~20 mM) at room temperature and 500 ppm spectral width. C, H, and N analyses were performed by the microanalytical service of the N.I.O.C. Research Institute of Petroleum Industry.

2.1. *Synthesis of Schiff bases*

2.1.1. Synthesis of H_2L^1. A mixture of pyrrole-2-carbaldehyde (0.46 g, 5 mmol) and 2-amino-4-chlorophenol (0.72 g, 5 mmol) was stirred in ethanol (20 mL) at 50°C for 3 h.

The product precipitated as yellow needles after standing overnight at room temperature. Crystals were collected, washed with ethanol, and dried over $CaCl_2$. Yield: 0.91 g (85%); m.p. 133–135°C; FT-IR (KBr, cm^{-1}): ν(C=N), 1624. ^{1}H NMR ($CDCl_3$): $\delta = 6.38$ (dd, $J = 3.7$, 2.6 Hz, 1H, H_e), 6.80 (dd, $J = 3.7$, 1.2 Hz, 1H, H_d), 6.92 (d, $J = 8.6$ Hz, 1H, H_c), 7.09 (not-resolved, 1H, H_f), 7.10 (dd, $J = 8.6$, 2.4 Hz, 1H, H_b), 7.21 (d, $J = 2.4$ Hz, 1H, H_a), 8.42 (s, 1H, CH=N), 9.40 (s, br, 1H, OH).

2.1.2. Synthesis of H_2L^2. 2-Hydroxy-1-naphtaldehyde (0.86 g, 5 mmol) and 2-amino-4-chlorophenol (0.72 g, 5 mmol) were stirred in ethanol (20 mL) at 0°C for 1 h, during which an orange precipitate gradually formed. The solid was filtered off, washed with ethanol and dried over $CaCl_2$. Yield: 0.43 g (65%); m.p. 205–207°C; ν(C=N), 1635. ^{1}H NMR (DMSO): $\delta = 6.59$ (d, $J = 9.3$ Hz, 1H, H_2), 6.66 (d, $J = 8.6$ Hz, 1H, H_c), 6.70 (dd, $J = 8.6$, 1.7 Hz, 1H, H_b), 6.97 (t, $J = 7.5$ Hz, 1H, H_4), 7.18 (t, $J = 7.5$ Hz, 1H, H_5), 7.26 (d, $J = 1.7$ Hz, 1H, H_a), 7.33 (d, $J = 7.5$ Hz, 1H, H_3), 7.41 (d, $J = 9.2$ Hz, 1H, H_1), 7.86 (d, $J = 8.3$ Hz, 1H, H_6), 9.04 (d, $J = 7.7$ Hz, 1H, CH=N), 9.66 (s, H_d), 15.17 (d, $J = 7.5$ Hz, 1H, H_e).

2.1.3. Synthesis of HL^3. 2-Hydroxy-1-naphtaldehyde (1.72 g, 10 mmol) and 2-(aminomethyl)pyridine (1.02 mL, 10 mmol) in ethanol (20 mL) were refluxed for 4 h. Then the brown solution was transferred to a separating decanter and the product was extracted with n-hexane (3×20 mL). The combined extracts were evaporated to give the yellow product. Yield: 1.08 g (58%); m.p. 90–95°C; ν(C=N), 1627. ^{1}H NMR (DMSO): $\delta = 5.0$ (d, $J = 4.0$ Hz, 2H, CH_2), 6.74 (d, $J = 9.3$ Hz, 1H, H_2), 7.21 (t, $J = 7.4$ Hz, 1H, H_4), 7.35 (t, $J = 6.2$ Hz, 1H, H_5), 7.45 (m, 2H, $H_{b,d}$), 7.65 (d, $J = 7.8$ Hz, 1H, H_3), 7.75 (d, $J = 9.3$ Hz, 1H, H_1), 7.84 (t, $J = 7.7$ Hz, 1H, H_c), 8.10 (d, $J = 8.4$ Hz, 1H, H_6), 8.60 (d, $J = 4.6$ Hz, 1H, H_a), 9.30 (d, $J = 9.3$ Hz, 1H, CH=N), 14.32 (s, br, 1H, N–H $\cdots$ O).

2.2. *Synthesis of organotin(IV) complexes*

2.2.1. Synthesis of $[SnPh_2(L^1)]$ (1). To a hot methanolic solution (20 mL) of H_2L^1 (0.20 g, 1 mmol), triethylamine (2 mmol) was added with constant stirring. After stirring for 30 min, $SnPh_2Cl_2$ (0.34 g, 1 mmol) in methanol (20 mL) was added dropwise. The solution was stirred for 1 h. During this time an orange precipitate gradually formed. The product was collected, washed with methanol and dried in vacuum over $CaCl_2$. Yield: 0.144 g (75%); m.p. 198–200°C; Anal. Calcd for $C_{23}H_{17}N_2OClSn$: C, 56.1; H, 3.5; N, 5.7%. Found: C, 56.4; H, 3.5; N, 5.3%. FT-IR (KBr, cm^{-1}): ν(C=N), 1580, ν(Sn–O), 596, ν(Sn–N), 448. ^{1}H NMR ($CDCl_3$): $\delta = 6.70$ (dd, $J = 3.7$, 1.7 Hz, 1H, H_e), 7.01 (d, $J = 8.7$ Hz, 1H, H_c), 7.07 (dd, $J = 8.7$, 2.4 Hz, 1H, H_b), 7.18 (dd, $J = 3.7$, 0.83 Hz, 1H, H_d), 7.30 (d, $J = 2.4$ Hz, 1H, H_a), 7.52 (not-resolved, 1H, H_f), 7.40–7.43 (m, 6H, $H_{3,4,5}$ in $SnPh_2$), 7.70–7.73 [m, 4H, $H_{2,6}$ in $SnPh_2$, $^3J(^{119}Sn–^1H) = 85$ Hz], 8.59 [s, 1H, CH=N, $^3J(^{119}Sn–^1H) = 76$ Hz]. $^{119}Sn\{^1H\}$ NMR ($CDCl_3$): $\delta = -280.0$.

2.2.2. Synthesis of $[SnPh_2(L^2)]$ (2). Complex **2** was synthesized as described for **1** from H_2L^2 (0.28 g, 1 mmol) and the red precipitate was collected after 3 h. Yield: 0.178 g (80%); m.p. 228–230°C; Anal. Calcd for $C_{29}H_{20}NO_2ClSn$: C, 61.2; H, 3.5; N, 2.5%.

Found: C, 61.3; H, 3.7; N, 2.3%. FT-IR (KBr, cm^{-1}): ν(C=N), 1617, ν(Sn–O), 554, ν(Sn–N), 427. ^{1}H NMR ($CDCl_3$): $\delta = 7.09$ (d, $J = 8.7$ Hz, 1H, H_c), 7.22 (dd, $J = 8.7$, 2.4 Hz, 1H, H_b), 7.28 (d, $J = 9.2$ Hz, 1H, H_2), 7.40–7.47 (m, 8H, H_4, H_a, $H_{3,4,5}$ in $SnPh_2$), 7.61 (t, $J = 7.7$ Hz, 1H, H_5), 7.78 (d, $J = 7.9$ Hz, 1H, H_3), 7.94 [m, 4H, $H_{2,6}$ in $SnPh_2$, $^3J(^{119}Sn–^1H) = 87.5$ Hz], 7.97 (d, $J = 9.2$ Hz, 1H, H_1), 8.08 (d, $J = 8.5$ Hz, 1H, H_6), 9.51 [s, 1H, CH=N, $^3J(^{119}Sn–^1H) = 60.6$ Hz]. $^{119}Sn\{^1H\}$ NMR ($CDCl_3$): $\delta = -321.2$.

2.2.3. Synthesis of [$SnMe_2(L^2)$] (3). H_2L^2 (0.28 g, 1 mmol) was dissolved in MeOH (20 mL) at 50–60°C and triethylamine (2 mmol) was added. The solution was stirred for 30 min and then $SnMe_2Cl_2$ (0.20 g, 1 mmol) in methanol (20 mL) was added. This solution was refluxed for 10 h. The orange product precipitated after reducing the solution volume at room temperature. The solid was filtered off, washed with methanol and dried in vacuum over $CaCl_2$. Yield: 0.194 g (65%); m.p. 223–225°C; Anal. Calcd for $C_{19}H_{16}NO_2ClSn$: C, 51.3; H, 3.6; N, 3.2%. Found: C, 51.5; H, 3.8; N, 3.2%. FT-IR (KBr, cm^{-1}): ν(C=N), 1617, ν_{as}(Sn–C), 574, ν_s(Sn–C), 551, ν(Sn–O), 513, ν(Sn–N), 443. ^{1}H NMR ($CDCl_3$): $\delta = 0.84$ [s, 6H, Sn–CH_3, $^2J(^{117/119}Sn–^1H) = 74.7/77.7$ Hz], 6.82 (d, $J = 8.7$ Hz, 1H, H_c), 6.95 (d, $J = 9.2$ Hz, 1H, H_2), 7.16 (dd, $J = 8.7$, 2.4 Hz, 1H, H_b), 7.40 (d, $J = 2.4$ Hz, 1H, H_a), 7.43 (t, $J = 7.5$ Hz, 1H, H_4), 7.64 (t, $J = 7.7$ Hz, 1H, H_5), 7.76 (d, $J = 7.4$ Hz, 1H, H_3), 7.88 (d, $J = 9.2$ Hz, 1H, H_1), 8.10 (d, $J = 8.4$ Hz, 1H, H_6), 9.45 [s, 1H, CH=N, $^3J(^{119}Sn–^1H) = 55.0$ Hz]. $^{119}Sn\{^1H\}$ NMR ($CDCl_3$): $\delta = -140.3$.

2.2.4. Synthesis of [$SnPhCl_2(L^3)$] (4). A solution of $SnPh_2Cl_2$ (0.34 g, 1 mmol) and HL^3 (0.20 g, 1 mmol) in benzene were refluxed for 15 h. The yellow product precipitated after reducing solution volume at room temperature. The solid was filtered off, washed with benzene and dried in vacuum over $CaCl_2$. Yield: 0.146 g (65%); m.p. 180–182°C; Anal. Calcd for $C_{23}H_{18}N_2OCl_2Sn$: C, 52.3; H, 3.4; N, 5.3%. Found: C, 51.9; H, 3.5; N, 4.9%. FT-IR (KBr, cm^{-1}): ν(C=N), 1627, ν(Sn–O), 540, ν(Sn–N), 475. ^{1}H NMR (DMSO): $\delta = 5.25$ [s, 2H, CH_2, $^3J(^{119}Sn–^1H) = 33.3$ Hz], 6.65 (d, $J = 9.1$ Hz, 1H, H_2), 7.01 (d, $J = 6.0$ Hz, $1H_{aromatic}$), 7.16–7.20 (m, $5H_{aromatic}$), 7.38 (d, $J = 8.1$ Hz, $2H_{aromatic}$), 7.51 (d, $J = 9.1$ Hz, 1H, H_1), 7.79–7.82 (m, 6H, $H_{3,4,5}$ in $SnPh_2$), 7.95 [dd, $J = 7.8$, 1.5 Hz, 4H, $H_{2,6}$ in $SnPh_2$, $^3J(^{119}Sn–^1H) = 130.0$ Hz], 9.04 [s, 1H, CH=N, $^3J(^{119}Sn–^1H) = 58.8$ Hz]. $^{119}Sn\{^1H\}$ NMR ($CDCl_3$): $\delta = -486.7$.

3. Results and discussion

Schiff bases used in this work, H_2L^1, H_2L^2, and HL^3, have been synthesized from reaction of pyrrole-2-carbaldehyde with 2-amino-4-chlorophenol, 2-hydroxy-1-naphtaldehyde with 2-amino-4-chlorophenol and 2-hydroxy-1-naphtaldehyde with 2-(aminomethyl)pyridine, respectively.

In general, aldimine Schiff bases exhibit tautomerism between the phenol-imine and the keto-amine forms and have two types of intramolecular hydrogen bonds (N–H···O or N···H–O). In the solid state, salicylaldimine and naphthaldimine Schiff bases tend to form the (N···H–O) and (N–H···O), respectively [26]. In solution, both types of hydrogen bonds have been observed indicating phenol-imine, O–H···N,

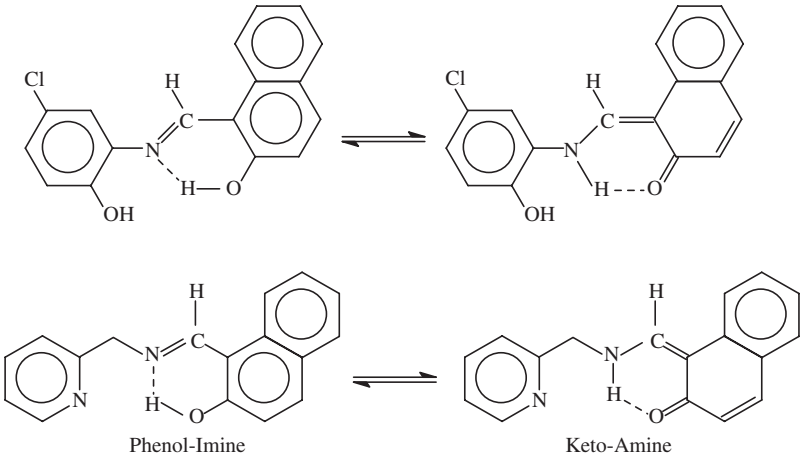


Figure 2. Tautomeric equilibria for H_2L^2 and HL^3.

and keto-amine, O···H–N, tautomerism [27]. Figure 2 shows this tautomeric equilibria for H_2L^2 and HL^3. The positions of the keto-phenol or amine-imine equilibrium and nature of the hydrogen bond in the six-membered chelate ring have been investigated by spectroscopic methods.

Complexes **1**–**4** were prepared by reaction of $SnMe_2Cl_2$ or $SnPh_2Cl_2$ with Schiff bases in the presence of triethylamine (for **1**–**3**) or in absence of it (for **4**). The composition of complexes was confirmed by their elemental analysis. In **1**–**3** chlorides were substituted by dianionic Schiff-base ligand. However, in **4** one phenyl group was removed as benzene and the monoanionic ligand substituted. Substitution of organic groups is rare in organotin(IV) reactions. Characterization of ligands and complexes and nature of bonding in complexes were examined by spectroscopic investigations.

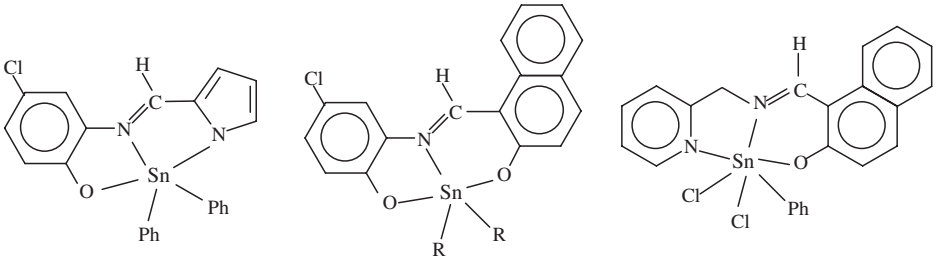
3.1. *IR spectra*

The azomethine C=N band which appears at 1625–1635 cm^{-1} in IR spectra of free ligands shifts to lower wavenumber in the complexes, indicating that the imine nitrogen atom is involved in coordination. New bands in the range ~400–600 cm^{-1} may be assigned to ν(Sn–O) and ν(Sn–N), supporting bonding of oxygen and nitrogen to tin [17, 18, 28, 29]. The presence of both ν_s(Sn–C) and ν_{as}(Sn–C) in the IR spectrum of **3** is consistent with a nonlinear Me–Sn–Me configuration.

3.2. *^{1}H NMR spectra*

In ^{1}H NMR spectra of Schiff bases the signals of CH=N in H_2L^2 and HL^3, CH_2 in HL^3 and H_e in H_2L^2 were doublets and hydroxyl of HL^3 was broadened. These observations support the location of the hydrogen on nitrogen and HCNH coupling for the keto-amine form of H_2L^2 and HL^3 (figure 2) in solution. In spectra of the complexes, the absence of signals due to phenolic protons suggests deprotonation and coordination of anions to tin.

A change in δ of azomethine proton resonance in ^{1}H NMR spectra of complexes relative to free ligands and satellites around this signal due to $^3J(^{119}Sn–^1H)$ coupling



(**1**) R = Ph (**2**), R = Me (**3**) (**4**)

Figure 3. Suggested structures for **1**–**4**.

indicate ligation of azomethine nitrogen to tin [30, 31]. In the spectrum of **4** the hydrogen of the methylene group adjacent to the imine nitrogen is a singlet accompanied by ^{119}Sn satellites; this also supports coordination of azomethine nitrogen to metal.

In spectra of all complexes, signals due to pyrrole and pyridine rings show some shifts from the free ligands indicating participation of the nitrogen in coordination with tin.

^{1}H NMR spectra of diphenyltin complexes (**1**, **2**, and **4**) show a doublet attributable to $H_{2,6}$ of Ph_2Sn. This signal has ^{119}Sn satellites due to ^{1}H–^{119}Sn coupling with $^{3}J(^{119}\text{Sn}–^{1}\text{H})$ larger than uncomplexed $SnPh_2Cl_2$ (81.7 Hz), in particular in **4**. Generally on complexation the magnitude of $J(^{119}\text{Sn}–^{1}\text{H})$ increases depending on the stereochemistry at tin and on the nature of the ligand; larger coupling constant indicates higher coordination number of tin [32].

The ^{1}H NMR spectrum of **3** shows a singlet at 0.84 ppm for $SnMe_2$ accompanied by satellites; $^{2}J(^{117/119}\text{Sn}–^{1}\text{H})$ is larger than for $SnMe_2Cl_2$ [$^{2}J(^{117/119}\text{Sn}–^{1}\text{H}) = 65.7/68.7$ Hz] and falls in the range for five-coordinate dimethyltin(IV) species [17, 33]. Substitution of $^{2}J(^{119}\text{Sn}–^{1}\text{H})$ in the Lockhart-Manders equation [34] gives a value of 128.0° for the Me–Sn–Me angle. Therefore in solution, similar to the solid phase, $SnMe_2$ is not linear. This angle is consistent with a trigonal bipyramidal structure with two methyl groups in equatorial positions and two electronegative oxygens axial (Bent's rule [35]).

3.3. *^{119}Sn NMR spectra*

The ^{119}Sn{^{1}H}NMR spectra of all complexes show one sharp singlet significantly at lower frequency than the original $SnMe_2Cl_2$ (+137 ppm) and $SnPh_2Cl_2$ (−32 ppm) [32]. ^{119}Sn chemical shifts are influenced by variation in the coordination number and bond angles at tin, by any dπ–pπ bonding effect, and by the presence of electronegative substituents. When the tin coordination number increases the ^{119}Sn signal moves to lower frequency. On the basis of the chemical shift ranges for organotin(IV) derivatives [36–39], it appears reasonable to assume that the coordination number of tin is five for **1**–**3** and six for **4**.

4. Conclusion

Diorganotin dichlorides react with Schiff bases, H_2L^1, H_2L^2, and HL^3, in the 1 : 1 ratio. In the complexes Schiff bases are completely deprotonated and coordinated tridentate

via phenolic oxygen, pyrrolic, and imine nitrogens in **1**, two phenolic oxygens and imine nitrogen in **2** and **3**, and phenolic oxygen, imine and pyridine nitrogens in **4** (figure 3). In view of the biological activity of various diorganotin(IV) complexes, specially with nitrogen donor ligands, these new complexes may have applications as medicinal or biocidal agents. These studies also show a further aspect of the structural chemistry of organotin complexes with multidentate Schiff-base ligands.

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