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TRIORGANOTIN(IV) DERIVATIVES OF BIDENTATE SCHIFF BASES: SYNTHESIS AND SPECTRAL STUDIES

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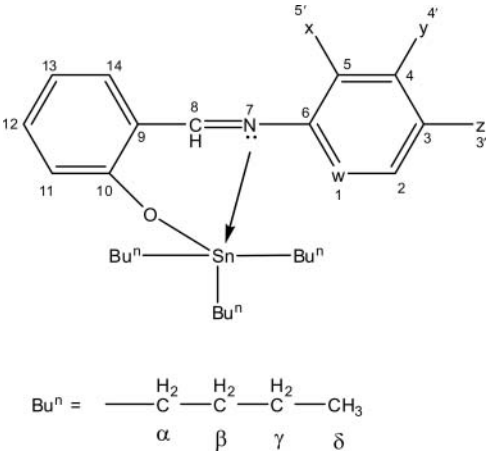
GRAPHICAL ABSTRACT



Abstract *Reaction of tri-n-butyl tin(IV) chloride with the sodium salt of Schiff bases [salicylidene-2-aminopyridine (sapH), salicylidene-2-amino-4-picoline (sapicH), salicylidene-2-methyl-1-aminobenzene (o-smabH), salicylidene-4-methyl-1-aminobenzene (p-smabH), salicylidene-1- aminobenzene (sabH), salicylidene-3-nitro-1-aminobenzene (snabH)] in MeOH-C_6H_6 mixture in 1:1 molar ratio produced complexes of the type [$Bu^n_3Sn(sb)$] (where sb = Schiff bases). All complexes obtained were characterized by elemental analysis (C, H, N, and Sn), infrared (IR), nuclear magnetic resonance (NMR; ^1H, ^{13}C, and ^{119}Sn), and TOF-MS spectroscopic studies. These complexes were found to be monomeric, colored viscous liquids and are soluble in polar solvents (methanol, ethanol, DMSO, and DMF). On the basis of ^{119}Sn NMR observations, a five coordination geometry around tin(IV) atom in these complexes is proposed tentatively.*

Keywords Schiff bases; synthesis; tri-n-butyl tin(IV); ^{119}Sn NMR; TOF-MS

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INTRODUCTION

Organotin(IV) complexes containing Schiff bases have received considerable attention in the field of bioinorganic chemistry[1,2] and in some cases have been found to be more effective specially to cleave the DNA.[3,4] Apart from this, organotin(IV) complexes have also been reported in the application of catalyst[5] and nonlinear optics.[6,7] Recently triorgnotin(IV) complexes display a higher biological activity[8] than their di- and mono-organotin(IV) analogus due to their binding ability with proteins. Instead of Schiff base ligands, benzyl bis (benzoylhydrazone) when reacted with di- and tri-organotin(IV) chlorides, yielded luminescent organotin(IV) complexes.[9] However, organotin(IV) complexes with Schiff base ligands derived from salicyldehyde have not been studied for the purpose of nonlinear optics and electroluminescent materials.[10–12] In view of this, we therefore describe herein the chemistry and spectral (IR, ^{1}H, ^{13}C, ^{119}Sn NMR, and TOF-MS) studies of organotin(IV) Schiff base complexes derived from salicyldehyde Schiff base ligands coordinating through an oxygen of the hydroxyl group and a nitrogen of the azomethine group.

RESULTS AND DISCUSSION

Tri-*n*-butyltin(IV) chloride reacted with the sodium salt of Schiff base ligands in an equimolar ratio and led to the formation of complexes (1–6), which are presented in Scheme 1.

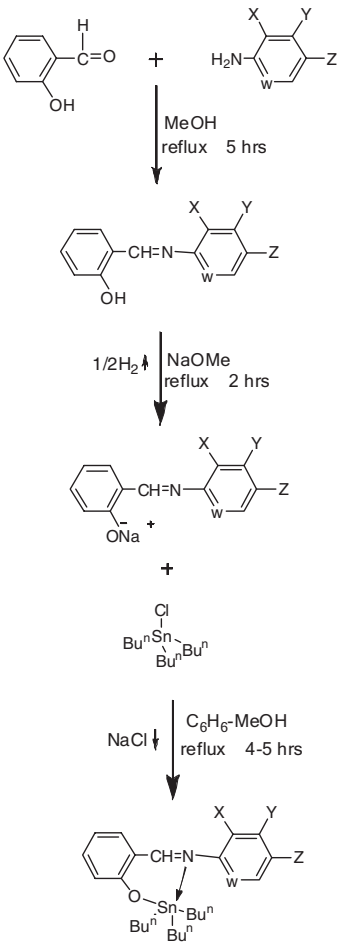
All these reactions were found to be quite facile and were completed within 4–5 h of refluxing in MeOH-C_6H_6 (30:20mL) solvent. The resulting complexes were obtained in good yields (66–73%). All these products are yellowish colored viscous liquid and were soluble in methanol, ethanol, isopropanol, chloroform, DMSO, and DMF.

IR Spectra

Selected IR data for all organotin(IV) complexes are reported in the experimental section. The IR spectra of the ligands exhibited the stretching vibration of the phenolic (O-H) group in the region 3484–3350 cm^{-1} which had disappeared from the spectra of the complexes.[13] This is assigned to the deprotonation of the phenolic oxygen atom of the ligand upon complexation with the tin atom. This was also confirmed by the appearance of the characteristic ($\nu_{C\text{-}O}$) phenolic band in 1294–1279 cm^{-1} region and a band at 549–532 cm^{-1} was observed region due to Sn—O stretching vibration.[14,15] In all the complexes, the ν(C=N) band occurring between 1612 and 1601 cm^{-1} is considerably shifted toward lower frequencies with respect to the free Schiff bases 1638–1621cm^{-1} indicating coordination of the azomethine nitrogen.[16] This was further supported by the appearance of a band at 470–458 cm^{-1} due to Sn—N stretching vibration.[17]

NMR Spectra

The absence of the phenolic (—OH) signal at 13.25–12.40 ppm in the complexes indicates the deprotonation of the —OH group.[18] The chemical shift value of the azomethine (CH=N) proton resonance exhibits signals at 9.45–8.49 ppm as singlets, similar to those reported in the literature.[19,20] The *n*-butyl group showed a well defined triplet in the range



Complex	W	X	Y	Z
1	N	H	H	H
2	N	H	$-CH_3$	H
3	C	$-CH_3$	H	H
4	C	H	H	$-CH_3$
5	C	H	H	H
6	C	H	$-NO_2$	H

Scheme 1 General method for preparation of Schiff bases and tin(IV) complexes.

0.96–0.91 ppm and a broad multiplet in the range 1.85–1.05 ppm owing to the terminal $-CH_3$ of the long alkyl chain and the $(-CH_2-)_3$ chain, respectively.[21,22] (Supplemental Materials Figures S 1 and S 2 available online.)

^{13}C NMR spectra of the Schiff bases containing azomethine carbon, signals observed in the range 159.9–156.5 ppm, which was shifted toward the lower value in the range 158.3-155.0 ppm in the all complexes, indicates the coordination of azomethine nitrogen to tin atom.[16,23] Whereas the Schiff bases containing phenolic carbon gave signal in the range

151.5–148.2 ppm characteristic feature for hydroxyl group present in the Schiff bases, which was shifted toward the higher value in the range 162.3–160.3 ppm, indicates the bonding through the phenolic oxygen[16,23] to the tin atom due to the formation of Sn—O bond. (Supplemental Materials Figures S 3 and S 4 available online.)

^{119}Sn NMR spectra can be used as an indicator of coordination number of tin atom. ^{119}Sn chemical shifts also show variation with the change in coordination number. The ^{119}Sn NMR chemical shifts for all the organotin(IV) complexes (1–6) are given in experimental section. The signal of the ^{119}Sn spectra appears at −147.6 to −128.0 ppm indicating that the tin atoms are five-coordinated in the complexes[21] (1–6). (Supplemental Materials Figures S 5 and S 6).

Mass Spectra

The TOF-MS ES$^+$ spectral data[24] for the complexes [Bu^n_3Sn(sap)] (1) and [Bu^n_3Sn(o-smab)] (3) were recorded and different fragmentation pattern with m/z have been suggested[14,16,18] (Supplemental Materials Schemes S 1 and S 2 available online). The mass spectra (Figures S 7 and S 8) of complexes (1) and (3) were observed and exhibited the group of peaks due to the presence of various isotopes of tin. These complexes showed molecular ion peaks at m/z 488.3050(3.4) [($C_{24}H_{36}N_2OSn$); calculated mass = 488.1849] and 502.8197(9) [($C_{26}H_{39}NOSn$) calculated mass = 501.2053] for (1) and (3), respectively. In both of the spectra of the complexes (1) and (3) the base peak due to the formation of fragment $C_6H_5O^+$ observed at 93.0490(100) and 93.0428(100), respectively. The other important peaks were also observed at m/z 432.5422(77.3), 410.2259(16.3), 373.2142(13.6), 329.2277(34.2), 316.1685(22.8), 270.3657(17.4), 241.2161(21.8), 214.3657(48.9), and 120.1437(27.3) due to the formation of various radicals $^{\bullet}Bu^n$, $C_6H_6N^{\bullet}$, CHN, $C_6H_6O^{\bullet}$, $Sn^{\bullet}$ in complex (1), whereas complex (3) showed some prominent peaks at m/z 444.0856(8.3), 386.0049(6.4), 330.2142(9.1), 269.8347(10.9), 239.1570(14.5), 213.1474(57.7), and 120.0752(23.1) due to the formation of various radical $^{\bullet}Bu^n$, $C_6H_7^{\bullet}$, CHN, $C_6H_6O^{\bullet}$, $Sn^{\bullet}$.

CONCLUSION

Six triorganotin(IV) complexes containing bidentate Schiff bases have been synthesized by the reaction of of tri-*n*-butyltin(IV)-chloride with the sodium salts of Schiff bases in the mixture of MeOH-C_6H_6 in equimolar ratio. These complexes (1–6) have been characterized by elemental analyses (Sn, C, H, N) and spectroscopic FT-IR, (^{1}H, ^{13}C and ^{119}Sn) NMR data as well as TOF-MS spectral study. The composition of the complexes has been determined on the basis of mass fragmentation pattern. On the basis of these analytical, spectral, and ^{119}Sn NMR chemical shift value, a five coordinated trigonal bipyramidal geometry[27] around tin(IV) atom proposed.

EXPERIMENTAL

Materials

All chemicals and reagents were of analytical grade quality. Tri-*n*-butylorganotin(IV) chloride (Aldrich), 2-aminopyridine (*M*erck), *o*-toludine (BDH), *p*-toludine (*M*erck),

salicyldehyde, *m*-nitroaniline, aniline, and 2-amino-4-picoline from (Loba) were used without further purifiacation. The solvents were dried before use according to the prescribed procedure.[25] The Schiff bases were synthesized by stirring a 1:1 molar ratio mixture of salicyldehyde and 2-aminopyridine, o-toludine, p-toludine, m-nitroaniline, aniline, and 2-aminopicoline in refluxing methanol according to the literature method.[14,16,18,26]

Physical Measurement

Elemental analyses were performed with a Haraeous Carlo Erba 1108 elemental analyzer. Tin was estimated gravimetrically as tin oxide SnO_2. Infrared spectra were recorded on a Perkin elmer 1000 FT-IR spectrometer in the range 4000–400 cm^{-1}. ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ on a JEOL–DELTA2 500 model spectrometer.^{119}Sn NMR spectra was recorded in DMSO-d_6 on Bruker DRX-300 spectrometer. The chemical shifts were reported in ppm relative to tetramethylsilane (TMS) for ^{1}H and ^{13}C, and tetramethyltin for ^{119}Sn shifts. Mass spectra were recorded on WATERS TOF MS spectrometer.

Synthesis of Complexes

All organotin(IV) complexes(1–6) of the type [$Bu^{n}_{3}Sn(sb)$] were synthesized by using same procedure as follows: A methanolic solution of the sodium salt of the schiff base sb(Na) (prepared by dissolving an equimolar amount of sodium metal and Schiff base (sbH) in ∼MeOH (30 ml) followed by the reflux of ∼2 h) was added dropwise to$Bu^{n}_{3}SnCl$ (∼20 ml benzene) with constant stirring in 1:1 molar ratios. The reaction mixture was allowed to reflux for 4–5 h. The precipitated NaCl was removed by filtration. The filtrate was concentrated removing the excess of solvent by distillation. The product was dried under reduced pressure.

Salicylidene-2-aminopyridene (sapH) orange solid; m.p.: 65 °C. Yield: 75%; Anal. Found: C, 72.58; H, 4.85; N, 13.93%. Calc. For $C_{12}H_{10}N_2O$: C, 72.71; H, 5.08; N, 14.13%. IR (KBr pellets, cm^{-1}): 3430($\nu_{O\text{-}H}$), 1634($\nu_{C=N}$), 1269($\nu_{C\text{-}O}$). ^{1}H NMR ($CDCl_3$, δ_H): 13.25(H, s, Ph-OH), 9.23(H, s, CH=N), 6.63–8.20(m, 8H, Ar-H, Py-H) ppm. ^{13}C NMR ($CDCl_3$, δ_C): 149.7 (s, CO, C-10), 158.3 (s, CN, C-8), 139.2 (C-2), 134.2 (C-3), 134.5 (C-4), 112.5 (C-5), 142.6 (C-6), 117.0 (C-9), 123.3 (C-11), 119.7 (C-12), 121.2 (C-13), 130.4 (C-14) ppm.

Salicylidene-2-amino-4-picoline (sapicH) orange solid; m.p.: 95 °C. Yield: 72%; Anal. Found: C, 73.33; H, 5.48; N, 12.98%. Calc. For $C_{13}H_{12}N_2O$: C, 73.56; H, 5.70; N, 13.20;%. IR (KBr pellets, cm^{-1}): 3436 ($\nu_{O\text{-}H}$), 1638 ($\nu_{C=N}$), 1267($\nu_{C\text{-}O}$). ^{1}H NMR ($CDCl_3$, δ_H): 13.18(H, s, Ph-OH), 9.37(H, s, CH=N), 6.63-8.20 (m, 7H, Ar-H, Py-H), 2.30 (s, 3H, Py-CH_3) ppm. ^{13}C NMR ($CDCl_3$, δ_C): 150.1 (s, CO, C-10), 158.7 (s, CN, C-8), 138.9 (C-2), 133.9 (C-3), 135.2 (C-4), 20.1 (C-4′), 111.0 (C-5), 141.9 (C-6), 116.7 (C-9), 125.8 (C-11), 118.8 (C-12), 120.6 (C-13), 129.8 (C-14) ppm.>

Salicylidene-2-methyl-1-aminobenzene (o-smabH) bright yellow crystal; m.p.: 59 °C. Yield: 73%; Anal. Found: C, 79.31; H, 5.98; N, 6.48%. Calc. For $C_{14}H_{13}NO$: C, 79.59; H, 6.20; N, 6.63%. IR (KBr pellets, cm^{-1}): 3484($\nu_{O\text{-}H}$), 1621($\nu_{C=N}$), 1272($\nu_{C\text{-}O}$). ^{1}H-NMR ($CDCl_3$, δ_H): 12.89(H, s, Ph-OH), 8.35(H, s, CH=N), 6.91–7.38(m, 8H, Ar-H), 2.38(3H, s, Ar-CH_3) ppm. ^{13}C NMR ($CDCl_3$, δ_C): 151.5 (s, CO, C-10), 157.5 (s, CN, C-8), 117.2 (C-1), 126.8 (C-2), 126.9 (C-3), 132.4 (C-4), 130.6 (C-5), 18.1 (C-5′), 147.5 (C-6), 117.8 (C-9), 133.0 (C-11), 119.3 (C-12), 118.9 (C-13), 132.1(C-14) ppm.

Salicylidene-4-methyl-1-aminobenzene (p-smabH) bright yellow crystal; m.p.: 60 °C. Yield: 85%; Anal. Found: C, 79.43; H, 6.03; N, 6.45%. Calc. For $C_{14}H_{13}NO$: C, 79.59; H, 6.20; N, 6.63%. IR (KBr pellets, cm^{-1}): 3458($\nu_{O\text{-}H}$), 1616 ($\nu_{C=N}$), 1268($\nu_{C\text{-}O}$). ^{1}H-NMR ($CDCl_3$, δ_H): 12.76(H, s, Ph-OH), 8.23(H, s, CH=N), 6.82-7.26(m, 8H, Ar-H), 2.36(3H, s, Ar-CH_3) ppm. ^{13}C NMR ($CDCl_3$, δ_C): 151.1 (s, CO, C-10), 156.5 (s, CN, C-8), 119.2 (C-1, C-5), 129.9 (C-2, C-4), 132.8 (C-3), 18.5 (C-3′), 145.8 (C-6), 117.1 (C-9), 132.0 (C-11), 120.9 (C-12), 118.9 (C-13), 133.8(C-14) ppm.

Salicylidene-1-aminobenzene (sabH) yellow crystal m.p.: 52 °C. Yield: 75%; Anal. Found: C, 78.95; H, 5.49; N, 6.93%. Calc. For $C_{13}H_{11}NO$: C, 79.16; H, 5.62; N, 7.10%. IR (KBr pellets, cm^{-1}): 3426($\nu_{O\text{-}H}$), 1635($\nu_{C=N}$), 1262($\nu_{C\text{-}O}$). ^{1}H-NMR ($CDCl_3$, δ_H): 12.40(H, s, Ph-OH), 8.40(H, s, CH=N), 6.62–7.91(m, 9H, Ar-H) ppm. ^{13}C NMR ($CDCl_3$, δ_C): 150.8 (s, CO, C-10), 159.9 (s, CN, C-8), 118.6 (C-1, C-5), 126.6 (C-2, C-4), 130.2 (C-3), 142.7 (C-6), 110.4 (C-9), 137.2 (C-11), 112.4 (C-12), 117.3 (C-13), 131.8 (C-14) ppm.

Salicylidene-3-nitro-1-aminobenzene (snabH) yellow solid; m.p.: 121 °C. Yield: 78%; Anal. Found: C, 64.28; H, 3.97; N, 11.38%. Calc. For $C_{13}H_{10}N_2O_3$: C, 64.46; H, 4.16; N, 11.56%. IR (KBr pellets, cm^{-1}): 3350($\nu_{O\text{-}H}$), 1638($\nu_{C=N}$), 1261($\nu_{C\text{-}O}$). ^{1}H NMR ($CDCl_3$, δ_H): 12.67(H, s, Ph-OH), 8.44(H, s, CH=N), 6.61–7.79(m, 8H, Ar-H) ppm. ^{13}C NMR ($CDCl_3$, δ_C): 148.2 (s, CO, C-10), 159.7 (s, CN, C-8), 132.0 (C-1), 137.1 (C-2), 121.2 (C-3), 139.4 (C-4), 117.3 (C-5), 147.4 (C-6), 115.8 (C-9), 139.4 (C-11), 118.2 (C-12), 120.2 (C-13), 132.2 (C-14) ppm

[Bu^n_3Sn(sap)] (1): Yield: 74%; Anal. Found: C, 59.07; H, 7.36; N, 5.69; Sn, 24.28%. Calc. For $C_{24}H_{36}N_2OSn$: C, 59.16; H, 7.45; N, 5.75; Sn, 24.36%. IR (KBr pellets, cm^{-1}): 1608 ($\nu_{C=N}$), 1294($\nu_{C\text{-}O}$), 549($\nu_{Sn\text{-}O}$), 519($\nu_{Sn\text{-}C}$), 461($\nu_{Sn\text{-}N}$). ^{1}H NMR ($CDCl_3$, δ_H): 9.43(H, s, CH=N), 6.56–7.96(m, 8H, Ar-H, Py-H), 1.05–1.81(m, 18H, $(CH_2)_3$-Sn), 0.94(t, 9H, H_3C-, $^3J_{H\text{-}H} = 7.4$ Hz) ppm. ^{13}C NMR ($CDCl_3$, δ_C): 161.7 (s, CO, C-10), 157.7 (s, CN, C-8), 138.9 (C-2), 130.7 (C-3), 138.6 (C-4), 109.8 (C-5), 145.5 (C-6), 113.7 (C-9), 133.2 (C-11), 119.9 (C-12), 132.2 (C-13), 132.4 (C-14), 26.7 (C-α), 27.3 (C-β), 27.2 (C-γ), 13.7 (C-δ) ppm. ^{119}Sn NMR (DMSO-d_6, δ_{Sn}): −140.8 ppm.

[Bu^n_3Sn(sapic)] (2): Yield: 71%; Anal. Found: C, 59.82; H, 7.56; N, 5.51; Sn, 23.59%. Calc. For $C_{25}H_{38}N_2OSn$: C, 59.90; H, 7.64; N, 5.59; Sn, 23.68%. IR (KBr pellets, cm^{-1}): 1610 ($\nu_{C=N}$), 1289($\nu_{C\text{-}O}$), 547($\nu_{Sn\text{-}O}$), 522($\nu_{Sn\text{-}C}$), 464($\nu_{Sn\text{-}N}$). ^{1}H NMR ($CDCl_3$, δ_H): 9.45(H, s, CH=N), 6.54-7.99 (m, 7H, Ar-H, Py-H), 2.32(s, 3H, Py-CH_3), 1.06-1.79(m, 18H, $(CH_2)_3$-Sn), 0.91(t, 9H, H_3C-, $^3J_{H\text{-}H} = 7.3$ Hz) ppm. ^{13}C NMR ($CDCl_3$, δ_C): 161.9 (s, CO, C-10), 157.6 (s, CN, C-8), 138.7 (C-2), 133.5 (C-3), 138.5 (C-4), 20.4 (C-4′), 109.5 (C-5), 146.1 (C-6), 113.8 (C-9), 133.9 (C-11), 119.9 (C-12), 122.6 (C-13), 133.8 (C-14), 26.7 (C-α), 27.3 (C-β), 27.1(C-γ), 13.6(C-δ) ppm. ^{119}Sn NMR (DMSO-d_6, δ_{Sn}): −128.0 ppm.

[Bu^n_3Sn(o-smab)] (3): Yield: 68%; Anal. Found: C, 62.38; H, 7.82; N, 2.77; Sn, 23.73%. Calc. For $C_{26}H_{39}NOSn$: C, 62.42; H, 7.86; N, 2.80; Sn, 23.73%. IR (KBr pellets, cm^{-1}): 1601 ($\nu_{C=N}$), 1292($\nu_{C\text{-}O}$), 532($\nu_{Sn\text{-}O}$), 512($\nu_{Sn\text{-}C}$), 470($\nu_{Sn\text{-}N}$). ^{1}H NMR ($CDCl_3$, δ_H): 8.58(H, s, CH=N), 6.95-7.41(m, 8H, Ar-H), 2.40(3H, s, Ar-CH_3), 1.37-1.85(m, 18H, $(CH_2)_3$-Sn), 0.96(t, 9H, H_3C-, $^3J_{H\text{-}H} = 7.1$ Hz) ppm. ^{13}C NMR ($CDCl_3$, δ_C): 161.3 (s, CO, C-10), 158.3 (s, CN, C-8), 117.3 (C-1), 126.9 (C-2), 127.1 (C-3), 132.2 (C-4), 130.8 (C-5), 18.3 (C-5′), 147.5 (C-6), 117.8 (C-9), 133.2 (C-11), 119.1 (C-12), 119.4 (C-13), 132.4 (C-14), 26.9 (C-α), 27.4 (C-β), 27.2 (C-γ), 13.7 (C-δ) ppm. ^{119}Sn NMR (DMSO-d_6, δ_{Sn}): −137.3 ppm.

[Bu^n_3Sn(p-smab)] (4): Yield: 66%; Anal. Found: C, 62.37; H, 7.69; N, 2.76; Sn, 23.65%. Calc. For $C_{26}H_{39}NOSn$: C, 62.42; H, 7.86; N, 2.80; Sn, 23.73%. IR (KBr pellets,

cm^{-1}): 1606 ($\nu_{C=N}$), 1284($\nu_{C\text{-}O}$), 538($\nu_{Sn\text{-}O}$), 516($\nu_{Sn\text{-}C}$), 468($\nu_{Sn\text{-}N}$). ^{1}H NMR ($CDCl_3$, δ_H): 8.57(H, s, CH=N), 6.93–7.41(m, 8H, Ar-H), 2.39(3H, s, Ar-CH_3), 1.12-1.84(m, 18H, $(CH_2)_3$-Sn), 0.93(t, 9H, H_3C-, $^3J_{H\text{-}H} = 7.2$ Hz) ppm. ^{13}C NMR ($CDCl_3$, δ_C): 162.3 (s, CO, C-10), 157.3 (s, CN, C-8), 126.9 (C-1, C-5), 127.1 (C-2, C-4), 133.9 (C-3), 18.3 (C-3′), 147.5 (C-6), 117.3 (C-9), 132.0 (C-11), 119.1 (C-12), 119.4 (C-13), 130.8 (C-14), 26.4 (C-α), 26.7 (C-β), 26.4 (C-γ), 13.6 (C-δ) ppm. ^{119}Sn NMR (DMSO-d_6, δ_{Sn}): −143.7 ppm.

[Bu^n_3Sn(sab)] (5): Yield: 70%; Anal. Found: C, 61.70; H, 7.61; N, 2.82; Sn, 24.35%. Calc. For $C_{25}H_{37}NOSn$: C, 61.75; H, 7.67; N, 2.88; Sn, 24.41%. IR (KBr pellets, cm^{-1}): 1604 ($\nu_{C=N}$), 1289($\nu_{C\text{-}O}$), 534($\nu_{Sn\text{-}O}$), 516($\nu_{Sn\text{-}C}$), 459($\nu_{Sn\text{-}N}$). ^{1}H NMR ($CDCl_3$, δ_H): 8.53(H, s, CH=N), 6.54–7.90(m, 9H, Ar-H), 1.24–1.77(m, 18H, $(CH_2)_3$-Sn), 0.95(t, 9H, H_3C-, $^3J_{H\text{-}H} = 7.3$ Hz) ppm. ^{13}C NMR ($CDCl_3$, δ_C): 162.0 (s, CO, C-10), 155.0 (s, CN, C-8), 119.8 (C-1, C-5), 127.1 (C-2, C-4), 119.8 (C-3), 144.0 (C-6), 112.4 (C-9), 136.7 (C-11), 113.6 (C-12), 117.8 (C-13), 129.3 (C-14), 26.4 (C-α), 26.7 (C-β), 26.5 (C-γ), 13.6 (C-δ) ppm. ^{119}Sn NMR (DMSO-d_6, δ_{Sn}): −136.9 ppm.

[Bu^n_3Sn(snab)] (6): Yield: 73%; Anal. Found: C, 56.48; H, 6.79; N, 5.21; Sn, 22.28%. Calc. For $C_{25}H_{36}N_2O_3Sn$: C, 56.52; H, 6.83; N, 5.27; Sn, 22.34%. IR (KBr pellets, cm^{-1}): 1612 ($\nu_{C=N}$), 1279($\nu_{C\text{-}O}$), 536($\nu_{Sn\text{-}O}$), 518($\nu_{Sn\text{-}C}$), 458($\nu_{Sn\text{-}N}$). ^{1}H NMR ($CDCl_3$, δ_H): 8.49(H, s, CH=N), 6.59–7.77(m, 8H, Ar-H), 1.27–1.68(m, 18H, $(CH_2)_3$-Sn), 0.96(t, 9H, H_3C-, $^3J_{H\text{-}H} = 7.3$ Hz) ppm. ^{13}C NMR ($CDCl_3$, δ_C): 160.3 (s, CO, C-10), 157.1 (s, CN, C-8), 132.4 (C-1), 136.4 (C-2), 122.3 (C-3), 140.3 (C-4), 116.6 (C-5), 148.6 (C-6), 118.8 (C-9), 138.2 (C-11), 119.4 (C-12), 119.6 (C-13), 133.5 (C-14), 26.1 (C-α), 26.3 (C-β), 26.1 (C-γ), 13.5 (C-δ) ppm. ^{119}Sn NMR (DMSO-d_6, δ_{Sn}): −147.6 ppm.

REFERENCES

1. Rehman, W.; Badshah, A.; Khan, S.; Tuyet, L. T. A. *Eur. J. Med. Chem.* **2009,** 44, 3981-3985.
2. Shujah, S.; Rehman, Z. U.; Muhammad, N.; Ali, S.; Khalid, N.; Tahir, M. N. *J. Organomet. Chem.* **2011,** 696, 2772-2781.
3. Prasad, K. S.; Kumar, L. S.; Chandan, S.; Jayalakshmi, B.; Revanasidappa, H. D. *Spectrochim. Acta A*, **2011,** 81, 276-282.
4. Rehman, W.; Baloch, M. K.; Badshah, A. *Eur. J. Med. Chem.* **2008,** 43, 2380-2385.
5. Darensbourg, D. J.; Ganguly, P.; Billodeaux, D. *Macromolecules* **2005,** 38, 5406-5410.
6. Reyes, H.; Garcia, C.; Farfan, N.; Santillan, R.; Lacroix, P. G.; Lepetit, C.; Nakatani, K. *J. Organomet. Chem.* **2004,** 689, 2303-2310.
7. Rivera, J. M.; Guzman, D.; Rodriguez, M.; Lamere, J. F.; Nakatani, K.; Santillan, R.; Lacroix, P. G.; Farfan, N. *J. Organomet. Chem.* **2006,** 691, 1722-1732.
8. Rehman, A. U.; Hussain, M.; Rehman, Z. U.; Ali, S.; Rauf, A.; Nasim, F. U. H.; Helliwell, M. *Inorg. Chim. Acta*, **2011,** 370, 27-35.
9. Torres, E. L.; Castillo, A. L. M.; Sanchez, J. F. F.; Mendiola, M. A. *J. Organomet. Chem.* **2010,** 695, 2305-2310.
10. Tang, C. W.; Slyke, S. A. V. *Appl. Phys. Lett.* **1987,** 51, 913.
11. Tao, X. T.; Shimoumura, M.; Suzuki, H.; Miyata, S.; Sasabe, H. *Appl. Phys. Lett.* **2000,** 76(24), 3522-3524.
12. Hamada, Y.; Sano, T. *J. Appl. Phys.* **1993,** 35, 511.
13. Kumar, P. K. *Indian J. Chem. A*, **1998,** 37A, 460-465.
14. Dubey, R. K.; Singh, A. P.; Dwivedi, N. *J. Indian Chem. Soc.* ***2011***, 88, 845-851.
15. Basu, S.; Gupta, G.; Das, B.; Rao, K. M. *J. Organomet. Chem.* **2010,** 695, 2098-2104.
16. Dubey, R. K.; Pathak, S. *Main Group Met. Chem.* **2008,** 31(1-2), 29-38.

17. Oztas, N. A.; Yenisehirli, G.; Ancin, N.; Oztas, S. G.; Ozcan, Y.; Ide, S. *Spectrochim. Acta A.* **2009,** 72, 929-935.
18. Dubey, R. K.; Pathak, S. *J. Indian Chem. Soc.* **2008,** 85, 53-58.
19. Shen, Y. Z.; Gu, H.; Pan, Y.; Dong, G.; Wu, T.; Jin, X. P.; Huang, X. Y.; Hu, H. *J. Organomet. Chem.* **2000,** 605, 234-238.
20. Yin, H. D.; Wang, Q. B.; Xue, S. C. *J. Organomet. Chem.* **2004,** 689, 2480-2485.
21. Rehman, S. U.; Shahid, K.; Ali, S.; Bhatti, M. H.; Parvez, M. *J. Organomet. Chem.* **2005,** 690, 1396-1408.
22. Rauf, M. K.; Saeed, M. A.; Din, I. U.; Bolte, M.; Badshah, A.; Mirza, B. *J. Organomet. Chem.* **2008,** 693, 3043-3048.
23. Belwal, S.; Saini, R. K.; Singh, R. V. *Indian J. Chem. A*, **1998,** 37A, 245-248.
24. Chandra, S.; Gautam, A.; Tyagi, M. *Trans. Met. Chem.* **2007,** 32, 1079-1084.
25. Armengo, W. L. F.; Chai, C. L. L. *Purification of Laboratory Chemicals, 6th ed.*; Elsevier: Burlington, MA, 2009.
26. Dubey, R. K.; Baranwal, P. *Main Group Met. Chem.* **2009,** 32, 321-339.
27. Nath, M.; Saini, P. K. *Dalton Trans.* **2011,** 40, 7077-7121.