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Synthesis and physicochemical characterization of some hexacoordinated tin(IV) Schiff base complexes

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Abstract

A freshly distilled tin(IV) tetrachloride interacted with sodium salts of Schiff bases in 1:2 molar ratios in a mixture of tetrahydrofuron (THF)-C_6H_6 produced complexes of the type [$SnCl_2(sb)_2$], [where sb=Schiff bases; salicylidene-2-aminopyridine (sap**H**) (**1**), salicylidene-2-amino-4-picoline (sapic**H**) (**2**), salicylidene-2-methyl-1-aminobenzene (*o*-smab**H**) (**3**), salicylidene-4-methyl-1-aminobenzene (*p*-smab**H**) (**4**), salicylidene-1-aminobenzene (sab**H**) (**5**), salicylidene-3-nitro-1-aminobenzene (snab**H**) (**6**)]. These complexes are soluble in polar solvents, monomeric, colored solids and characterized by elemental analysis (Sn, Cl, C, H, and N), spectral analysis [infrared and nuclear magnetic resonance (^{1}H, ^{13}C, and ^{119}Sn)], and time-of-flight mass spectrometry studies.

Keywords: hexacoordinated tin(IV) complexes; Schiff bases; ^{119}Sn NMR; synthesis; TOF-MS.

Introduction

Studies on inorganic and organometallic chemistry of tin(IV) metal provide fundamental information about Lewis acid-base model (Farcasiu et al., 2001; Denmark and Fu, 2003). Tin(IV) chloride is a strong Lewis acid-forming adduct with a variety of neutral ligands (Bacchi et al., 1998; Pettinari et al., 1998a). The pronounced dependence of stereochemistry of the complex formed depends on the nature of ligands containing P, O, N, and S donor atoms (Sousa et al., 1992; Barbieri et al., 1993; Sanhoury et al., 2008); Schiff base ligand coordinated to tin(IV) tetrachloride produced mixed ligand Schiff base complexes showing various coordination number around tin(IV) atoms. Such type of organotin(IV) complexes were found to be biologically potent (Shujah et al., 2010, 2011; Prasad et al., 2011). However, chlorinated species of 8-hydroxyquinoline has been proved as antibacterial and antifungal agents (Meyer et al., 1980); whereas chlorinated Sn(IV), Al(III), and Ti(IV) compounds used as catalyst for ring opening polymerization (Darensbourg et al., 2005; Lee et al., 2007). Keeping above facts in view we have decided to prepare various Schiff bases, containing 'O' and 'N' donor atoms, derived from salicyldehyde, which were further reacted with tin(IV) chloride forming new tin(IV) (-chloro-) Schiff base mixed ligand complexes. In this article, we report the synthesis of [$SnCl_2(sb)_2$] [where sb=Schiff bases; salicylidene-2-aminopyridine (sap**H**) (**1**), salicylidene-2-amino-4-picoline (sapic**H**) (**2**), salicylidene-2-methyl-1-aminobenzene (*o*-smab**H**) (**3**), salicylidene-4-methyl-1-aminobenzene (*p*-smab**H**) (**4**), salicylidene-1-aminobenzene (sab**H**) (**5**), salicylidene-3-nitro-1-aminobenzene (snab**H**) (**6**)] type of complexes. These complexes have been characterized by various spectroscopic [infrared (IR), nuclear magnetic resonance (^{1}H, ^{13}C, and ^{119}Sn NMR)] and time-of-flight mass spectrometry (TOF-MS) techniques and showed hexacoordinated geometry around tin(IV) atom.

Results and discussion

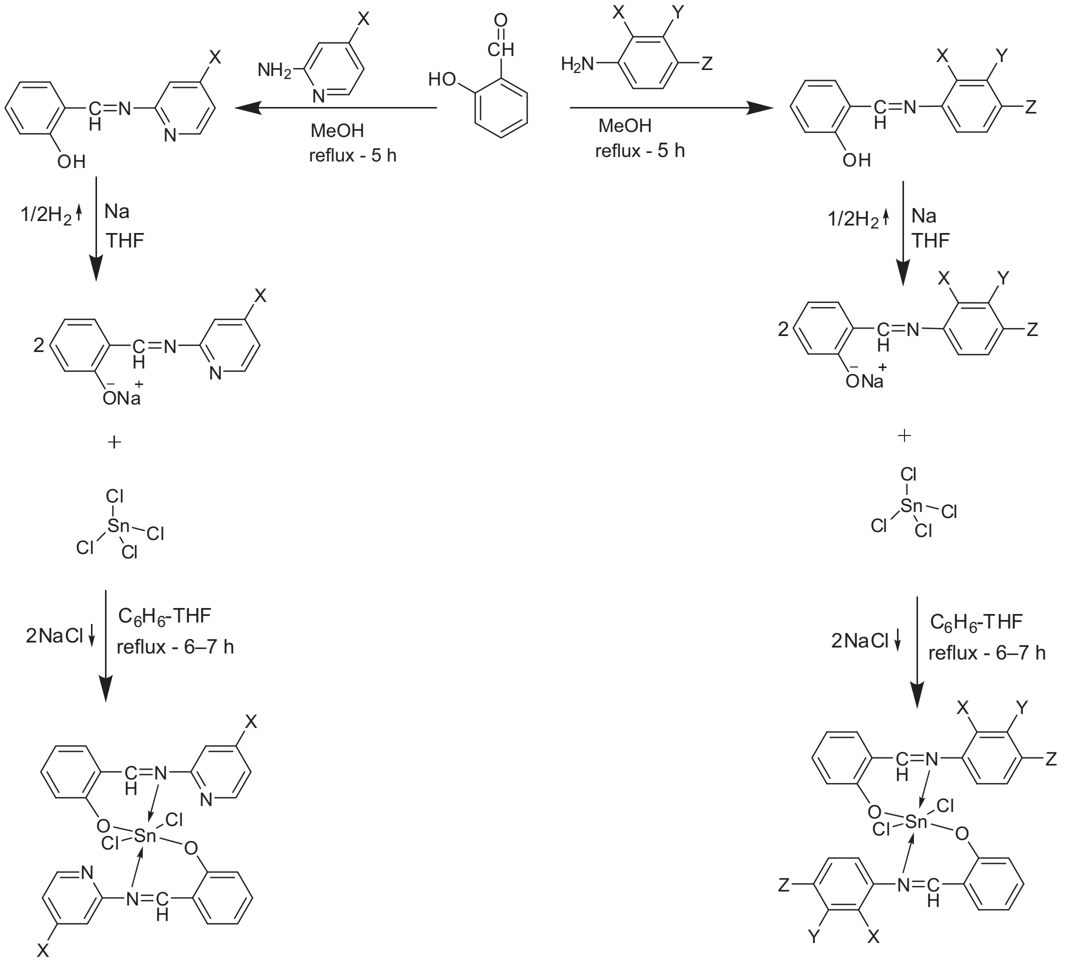
The new complexes were prepared in quantitative yields and are represented in Scheme 1. All these complexes are yellow- to brown-colored solids and were soluble in methanol, ethanol, isopropanol, chloroform, DMSO, and DMF.

Infrared spectra

Selected IR data for all tin(IV) complexes are reported in the experimental section. The salient features in IR studies are the disappearance of stretching vibration band due to phenolic (O-H) group in the region 3484–3350 cm^{-1}, which is assigned to the deprotonation of phenolic hydrogen on complexation (Kumar, 1998; Dubey et al., 2011). This change is also supported by the shifting of $\nu_{C\text{-}O}$ toward higher frequency scale (Dubey and Baranwal, 2009) and the appearance of new band in the region 540–532 cm^{-1} assignable to $\nu_{Sn\text{-}O}$ (Oztas et al., 2009). The coordination through azomethine nitrogen was supported by the shifting of $\nu_{C=N}$ (1624–1609 cm^{-1}) toward lower frequencies with respect to that observed (1638–1621 cm^{-1}) in free Schiff bases. The involvement of coordination of azomethine nitrogen was further supported by the appearance of a band in the 458–450 cm^{-1} region due to Sn-N stretching vibration (Dubey and Pathak, 2008a).

^{1}H nuclear magnetic resonance

The ^{1}H-NMR spectra of tin(IV) complexes exhibit an absence of signal due to the OH group in the region 13.25–12.40 ppm, which is indicative of metalation of the OH group. The appearance of azomethine hydrogen signals in the region 9.39–8.42



Complex	X
(**1**)	H
(**2**)	CH_3

Complex	X	Y	Z
(**3**)	CH_3	H	H
(**4**)	H	H	CH_3
(**5**)	H	H	H
(**6**)	H	NO_2	H

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

ppm supports the coordination of the nitrogen atom (Shen et al., 2000; Yin et al., 2004).

^{13}C nuclear magnetic resonance

The ^{13}C spectral data (given in experimental section) are also supportive of complex formation involving bonding through azomethine nitrogen and phenolic oxygen atom. For example, the observed signal in the region 157.88–154.88 ppm in the complex with respect to the corresponding free Schiff base ligands 159.92–156.51 ppm supports the coordination of the azomethine nitrogen to the tin atom (Belwal et al., 1998). The signals appearing in the region 151.56–148.20 ppm for free Schiff base ligands are shifted downfield in the complexes (Belwal et al., 1998) 161.32–160.24 ppm, which is indicative of bonding through phenolic oxygen and the formation of Sn-O bond.

^{119}Sn nuclear magnetic resonance

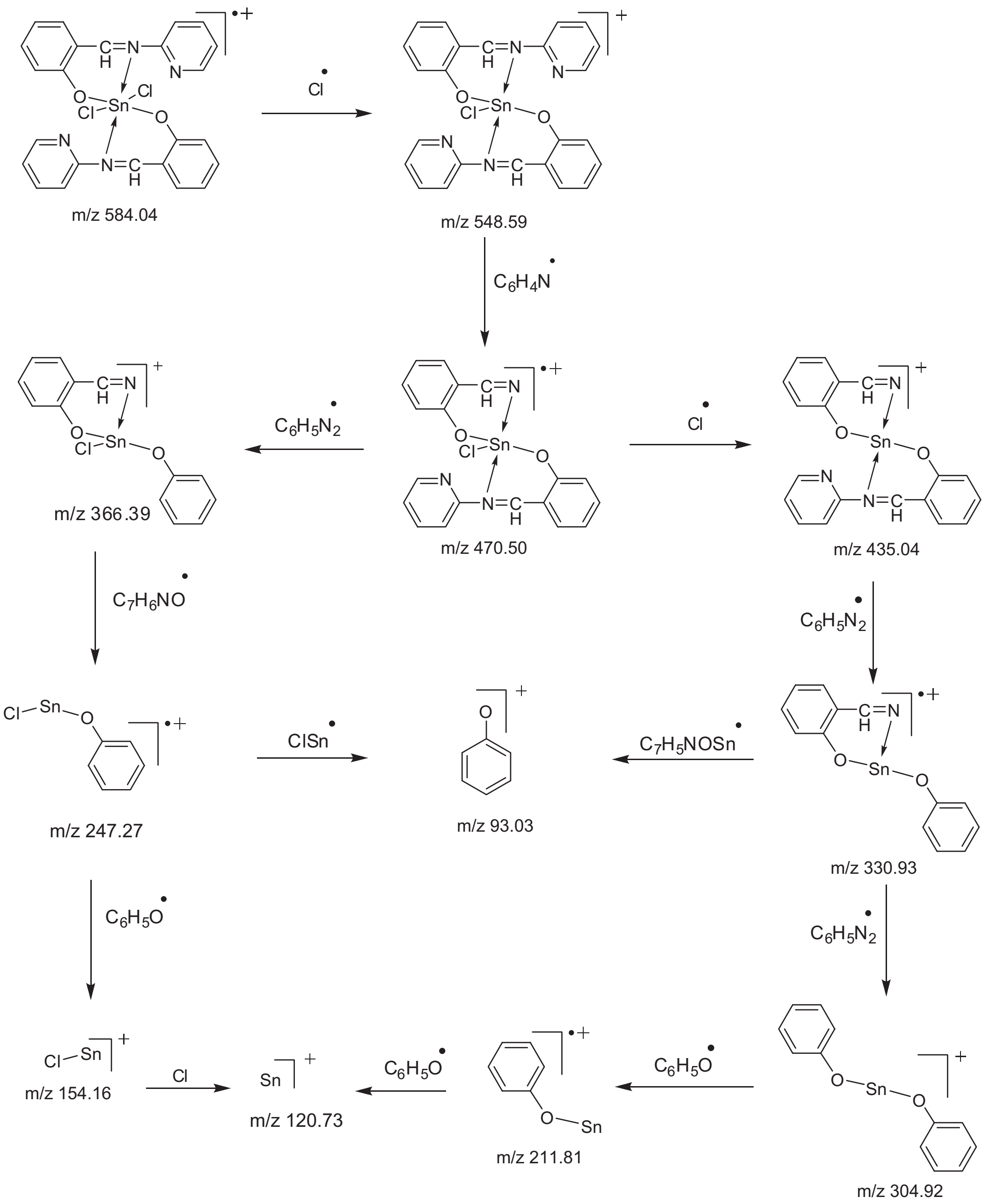
The observed ^{119}Sn NMR signals (in ppm) for complexes **1** (-611), **2** (-610) **3** (-619), **4** (-620), **5** (-608), **6** (-609) are supportive of the presence of the hexacoordinated tin(IV) center (Wrackmeyer, 1985; Pettinari et al., 1998b, 2001).

Time-of-flight mass spectrometry

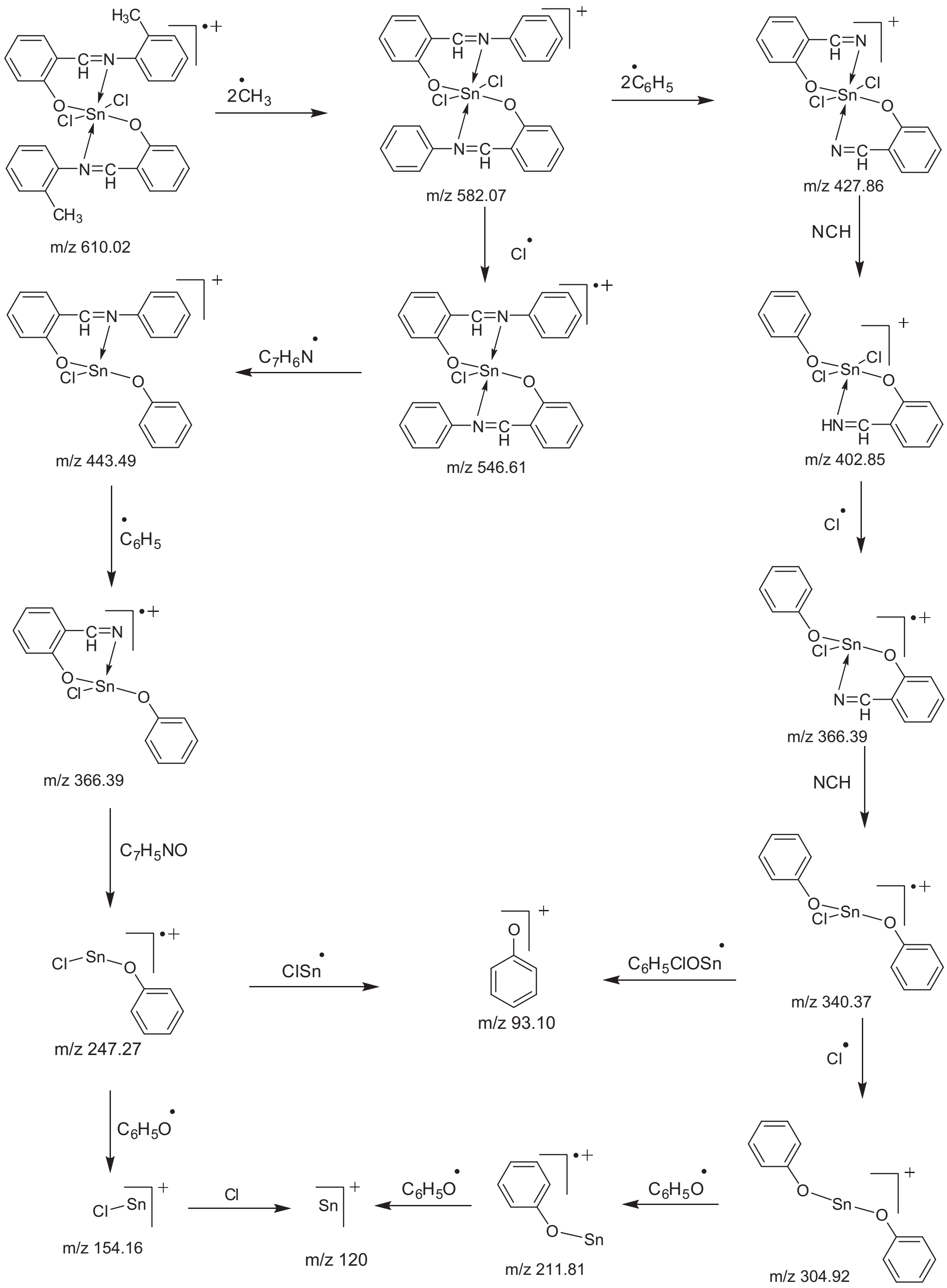
The TOF-MS ES$^+$ (Chandra et al., 2007) spectral data for the complexes $[SnCl_2(sap)_2]$ **(1)** and $[SnCl_2(o\text{-smab})_2]$ **(3)** were recorded, and different fragmentation patterns with *m/z* have been suggested (Shahzadi et al., 2006; Dubey and Pathak, 2008a,b) (Schemes 2 and 3). In the mass spectral data for complexes **(1)** and **(3)**, most of the fragment ions were observed in the form of group of peaks due to the presence of various isotopes of tin. In the above spectra (Figures 1 and 2) of the complexes, the molecular ion peaks are observed at *m/z* 581.18 [($C_{24}H_{18}Cl_2N_4O_2Sn$); calculated mass = 584.04] and 612.12 [($C_{28}H_{24}Cl_2N_2O_2Sn$), calculated mass = 610.02] for **(1)** and **(3)**, respectively. In both of the spectra, the base peak observed was due to the formation of fragment $[C_6H_5O]^+$ at *m/z* 93. Other important peaks were also observed at *m/z* 542.15, 472.05, 436.08, 367.17, 322.20, 303.16, 243.16, 212.12, 151.02, and 120.16 due to the formation of various radicals in complex **(1)**, whereas complex **(3)** showed some prominent peaks at *m/z* 582.37, 545.38, 442.22, 429.13, 403.30, 367.21, 347.22, 303.16, 245.21, 212.38, 149.11, and 120.06, corresponding to various fragments.

Conclusion

Six new complexes (**1–6**) of tin(IV) have been prepared and characterized spectroscopically, where all the ligands



Scheme 2 Fragmentation pattern of $[SnCl_2(sap)_2]$ **(1)**.



Scheme 3 Fragmentation pattern of [$SnCl_2$(*o*-smab)$_2$] **(3)**.

are having ‘O’ and ‘N’ donor atoms, of which complexes **1** and **2** contain heterocyclic ‘N’ atom along with phenolic ‘O’ and azomethine ‘N’ atom (i.e., three available bonding sites). It is tentatively proposed that all the six complexes exhibited hexacoordinated geometry around the tin(IV) atom.

Experimental

Materials

Analytical-grade chemicals and reagents were used. Tin tetrachloride was distilled under vacuum before use. Salicyldehyde (Loba

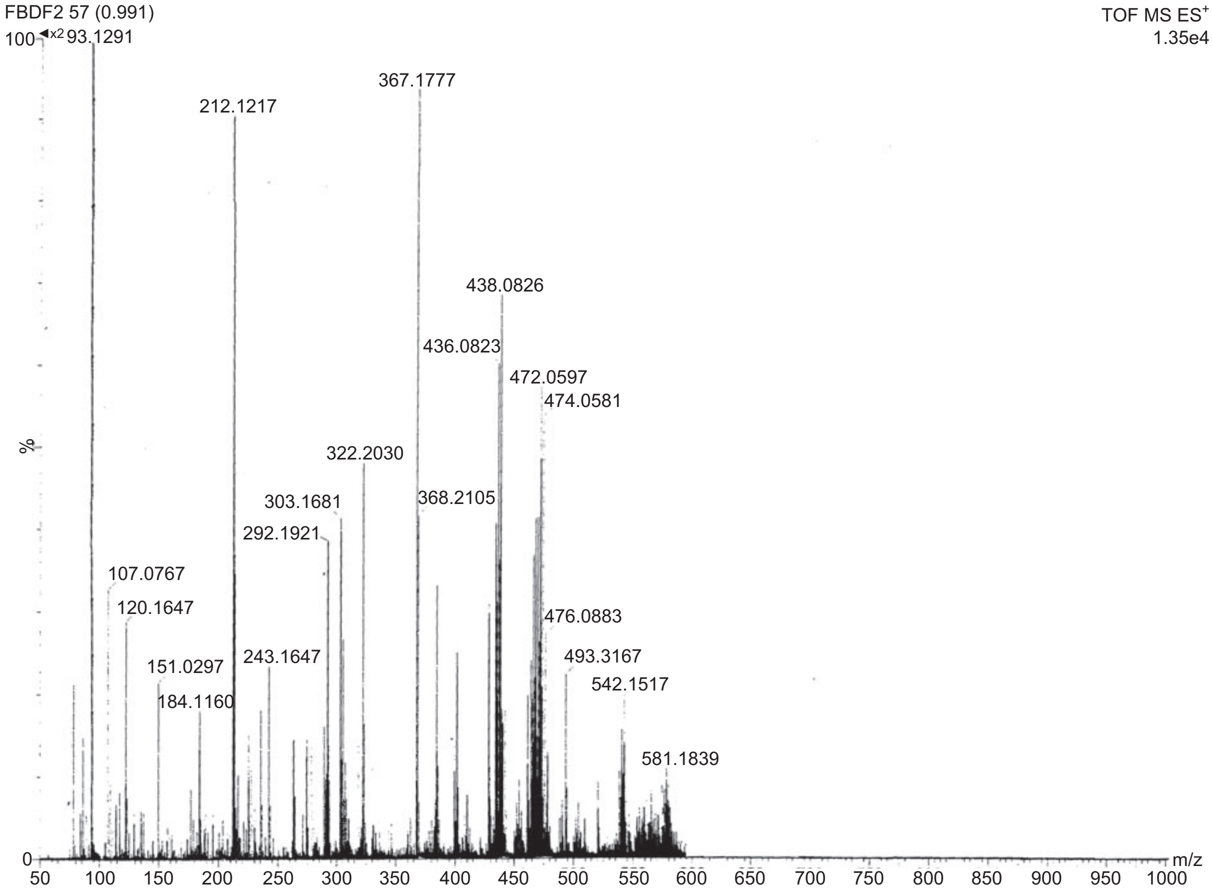


Figure 1 TOF-MS spectrum of $[SnCl_2(sap)_2]$ (**1**).

Chemie, Mumbai, India), 2-aminopyridine (Merck, Germany), *o*-toludine (BDH, UAE), *p*-toludine (Merck), *m*-nitroaniline (Loba), aniline (Loba), and 2-amino-4-picoline (Loba) were used without further purification. The solvents were dried before use according to the standard procedure (Perrin and Armengo, 1988). The Schiff base ligands were synthesized according to literature method (Dubey et al., 2005, 2008; Dubey and Baranwal, 2009).

Physical measurements

Elemental analyses for C, H, and N were performed on a Haraeus Carlo Erba 1108 elemental analyser (UK). Tin was estimated gravimetrically as SnO_2 after decomposition with concentrated HNO_3. IR spectra were recorded on a Perkin Elmer 100 (England) fourier transform infrared spectroscopy (FTIR) spectrophotometer in the range of 4000–400 cm^{-1}. NMR (^{1}H, ^{13}C, and ^{119}Sn) spectra (in a mixture of DMSO-d$_6$ and $CDCl_3$) were recorded on Bruker DRX-300 spectrometer (NMR-AI-300, Japan). Chemical shifts are given in parts per million relative to Me_4Si (^{1}H, ^{13}C) and Me_4Sn (^{119}Sn). Mass spectra were recorded on Waters TOF MS/MS spectrometer (USA).

Synthesis of complexes

Similar procedure was used for the preparation of the complexes (**1–6**); therefore, for the sake of brevity, general preparative details are given below. To a 20-ml benzene solution of $SnCl_4$, a THF solution of an appropriate sodium salt of Schiff base [prepared by dissolving equimolar amount of sodium metal and a Schiff base (HL) in 30 ml THF] was added dropwise in 1:2 molar ratio with constant stirring. The reaction mixture was allowed to reflux for 5 h. The precipitated NaCl was removed by filtration. The solvent was removed by distillation. The solid products were dried under reduced pressure and recrystallized from the mixture of THF-*n*-hexane solvent.

$[SnCl_2(sap)_2]$ (1) Yield: 84%; mp: 140°C–141°C. Anal. found: C, 49.16%; H, 2.93%; N, 9.13%; Cl, 11.87%; Sn, 20.06%. Calc. for $C_{24}H_{18}Cl_2N_4O_2Sn$: C, 49.36%; H, 3.11%; N, 9.59%; Cl, 12.14%; Sn, 20.33%. IR (KBr pellets, cm^{-1}): 1624($\nu_{C=N}$), 1288($\nu_{C\text{-}O}$), 540($\nu_{Sn\text{-}O}$), 458($\nu_{Sn\text{-}N}$). ^{1}H NMR ($CDCl_3$/DMSO-d$_6$, δ_H): 9.37(2H, s, CH=N), 8.45(2H, d, Py-H), 6.44–7.89(m, 14H, Ar-H, Py-H), ppm. ^{13}C NMR ($CDCl_3$/DMSO-d$_6$, δ_C): 160.44 (s, CO), 156.23 (s, CN), 147.76, 137.43, 132.64, 127.69, 127.07, 121.54, 118.87, 118.09, 117.69, 115.85 (s, Ar-C, Py-C), ppm. ^{119}Sn NMR ($CDCl_3$/DMSO-d$_6$) δ_{Sn}: -608, ppm.

$[SnCl_2(sapic)_2]$ (2) Yield: 81%; mp: 142°C–144°C. Anal. found: C, 50.82%; H, 3.46%; N, 8.81%; Cl, 11.23%; Sn, 19.09%. Calc. for $C_{26}H_{22}Cl_2N_4O_2Sn$: C, 51.2%; H, 3.62%; N, 9.15%; Cl, 11.58%; Sn, 19.39%. IR (KBr pellets, cm^{-1}): 1620($\nu_{C=N}$), 1286($\nu_{C\text{-}O}$), 537($\nu_{Sn\text{-}O}$), 451($\nu_{Sn\text{-}N}$). ^{1}H NMR ($CDCl_3$/DMSO-d$_6$, δ_H): 9.39(2H, s, CH=N), 8.46(2H, d, Py-H), 6.60–7.80(m, 12H, Ar-H, Py-H), 2.37(s, 6H, Py-CH_3), ppm. ^{13}C NMR ($CDCl_3$/DMSO-d$_6$, δ_C): 160.27 (s, CO), 156.08 (s, CN), 147.64, 137.38, 132.48, 127.79, 126.99, 121.47,

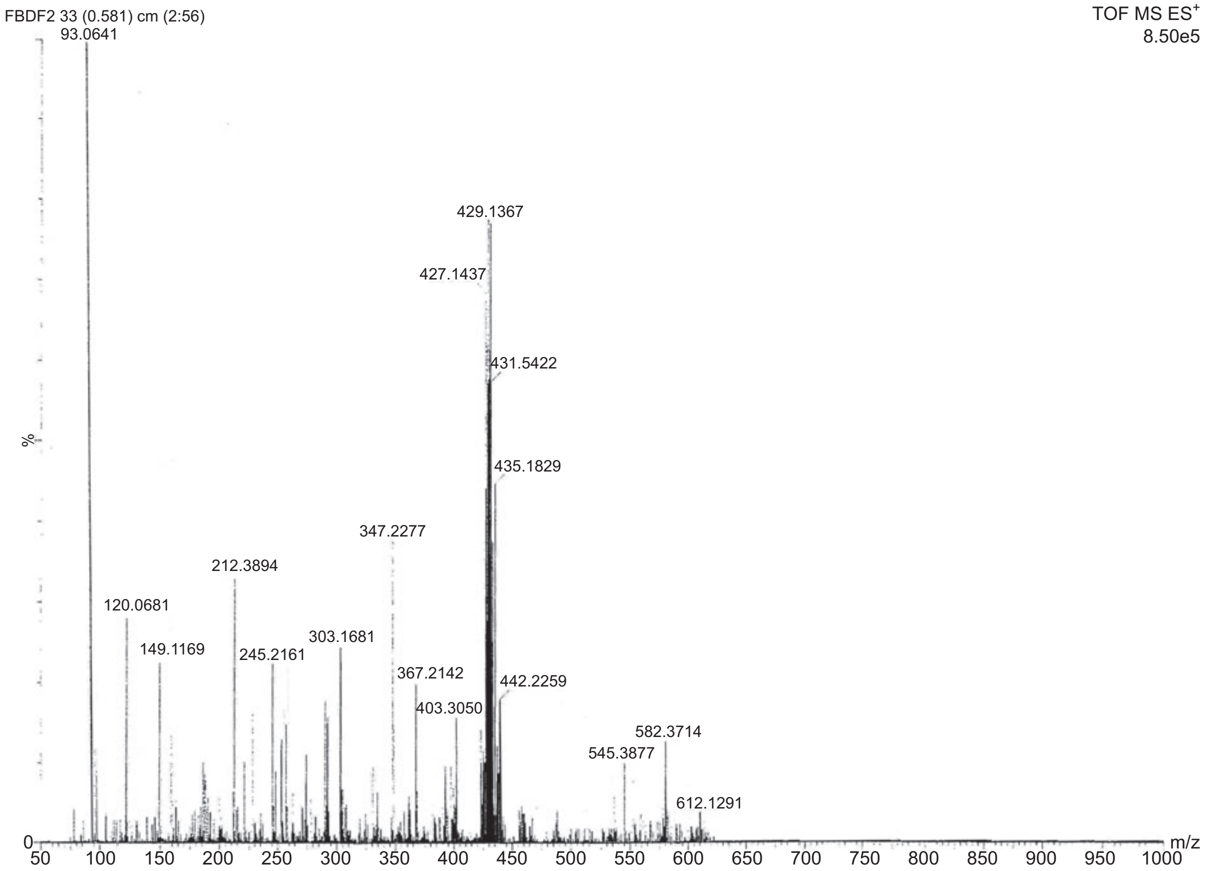


Figure 2 TOF-MS spectrum of $[SnCl_2(o\text{-smab})_2]$ **(3)**.

119.82, 118.99, 117.55, 115.99 (s, Ar-C, Py-C), 20.41 (s, Ar-CH_3), ppm. ^{119}Sn NMR ($CDCl_3$/DMSO-d_6) δ_{Sn}: -609, ppm.

$[SnCl_2(\boldsymbol{o}\text{-smab})_2]$ (3) Yield: 82%; mp: 144°C–146°C. Anal. found: C, 54.83%; H, 3.82%; N, 4.47%; Cl, 11.53%; Sn, 19.39%. Calc. for $C_{28}H_{24}Cl_2N_2O_2Sn$: C, 55.12%; H, 3.96%; N, 4.59%; Cl, 11.62%; Sn, 19.46%. IR (KBr pellets, cm^{-1}): 1609($\nu_{C=N}$), 1290($\nu_{C\text{-}O}$), 532($\nu_{Sn\text{-}O}$), 450($\nu_{Sn\text{-}N}$). ^{1}H NMR ($CDCl_3$/DMSO-d_6, δ_H): 8.58(2H, s, CH=N), 6.85–7.70(m, 16H, Ar-H), 2.35(6H, s, Ar-CH_3), ppm. ^{13}C NMR ($CDCl_3$/DMSO-d_6, δ_C): 159.46 (s, CO), 154.64 (s, CN), 145.64, 131.61, 130.40, 129.12, 128.88, 125.63, 125.28, 117.82, 117.56, 116.36, 115.33 (s, Ar-C), 16.61 (s, Ar-CH_3), ppm. ^{119}Sn NMR ($CDCl_3$/DMSO-d_6) δ_{Sn}: -611, ppm.

$[SnCl_2(\boldsymbol{p}\text{-smab})_2]$ (4) Yield: 78%; mp: 140°C–141°C. Anal. found: C, 54.92%; H, 3.78%; N, 4.42%; Cl, 11.49%; Sn, 19.35%. Calc. for $C_{28}H_{24}Cl_2N_2O_2Sn$: C, 55.12%; H, 3.96%; N, 4.59%; Cl, 11.62%; Sn, 19.46%. IR (KBr pellets, cm^{-1}): 1606($\nu_{C=N}$), 1284($\nu_{C\text{-}O}$), 538($\nu_{Sn\text{-}O}$), 459($\nu_{Sn\text{-}N}$). ^{1}H NMR ($CDCl_3$/DMSO-d_6, δ_H): 8.49(2H, s, CH=N), 6.57–7.32(m, 16H, Ar-H), 2.27(6H, s, Ar-CH_3), ppm. ^{13}C NMR ($CDCl_3$/DMSO-d_6, δ_C): 160.24 (s, CO), 154.88 (s, CN), 146.37, 132.49, 131.62, 129.96, 128.21, 127.30, 126.65, 118.48, 116.93 (s, Ar-C), 17.30 (s, Ar-CH_3), ppm. ^{119}Sn NMR ($CDCl_3$/DMSO-d_6) δ_{Sn}: -610, ppm.

$[SnCl_2(sab)_2]$ (5) Yield: 80%; mp: 136°C–138°C. Anal. found: C, 53.42%; H, 3.34%; N, 4.65%; Cl, 11.92%; Sn, 20.15%. Calc. for $C_{26}H_{20}Cl_2N_2O_2Sn$: C, 53.65%; H, 3.46%; N, 4.81%; Cl, 12.18%; Sn, 20.39%. IR (KBr pellets, cm^{-1}): 1604($\nu_{C=N}$), 1289($\nu_{C\text{-}O}$), 534($\nu_{Sn\text{-}O}$), 455($\nu_{Sn\text{-}N}$). ^{1}H NMR ($CDCl_3$/DMSO-d_6, δ_H): 8.42(2H, s, CH=N), 6.47–7.75(m, 18H, Ar-H), ppm. ^{13}C NMR ($CDCl_3$/DMSO-d_6, δ_C): 160.78 (s, CO), 156.54 (s, CN), 147.98, 137.57, 132.86, 132.50, 127.31, 121.68, 118.27, 117.95, 116.15 (s, Ar-C), ppm. ^{119}Sn NMR ($CDCl_3$/DMSO-d_6) δ_{Sn}: -619, ppm.

$[SnCl_2(snab)_2]$ (6) Yield: 76%; mp: 127°C–128°C. Anal. found: C, 46.21%; H, 2.44%; N, 8.02%; Cl, 10.12%; Sn, 17.35%. Calc. for $C_{26}H_{18}Cl_2N_4O_6Sn$: C, 46.47%; H, 2.70%; N, 8.34%; Cl, 10.55%; Sn, 17.66%. IR (KBr pellets, cm^{-1}): 1612($\nu_{C=N}$), 1279($\nu_{C\text{-}O}$), 536($\nu_{Sn\text{-}O}$), 456($\nu_{Sn\text{-}N}$). ^{1}H NMR ($CDCl_3$/DMSO-d_6, δ_H): 8.44(2H, s, CH=N), 6.55–7.77(m, 16H, Ar-H), ppm. ^{13}C NMR ($CDCl_3$/DMSO-d_6, δ_C): 161.32 (s, CO), 157.15 (s, CN), 148.64, 140.33, 138.29, 136.45, 133.54, 132.52, 122.40, 119.78, 119.49, 118.92, 116.73, (s, Ar-C), ppm. ^{119}Sn NMR ($CDCl_3$/DMSO-d_6) δ_{Sn}: -620, ppm.

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