

## Synthesis and Characterization of Some New Sn(II) Aromatic Schiff Base Complex

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Thirty two complexes of Sn(II) of the type  $\text{SnX}_2 \cdot \text{SB}$  (where  $\text{X} = \text{Cl}^-$ ,  $\text{Br}^-$ ; SB = Schiff base) derived from diamines and aromatic carbonyl compounds have been prepared and characterised by elemental analysis, conductivity measurements, IR and XPS data.

### INTRODUCTION

Although a large number of Sn(II) coordination compounds are known in the literature<sup>1</sup>, the limited number of Sn(II) compounds with aromatic Schiff base ligands are known<sup>2-6</sup>. We report here the synthesis of Sn(II) complexes with various bidentate Schiff base ligands derived from benzaldehyde, acetophenone, cinnamaldehyde and 2-naphthaldehyde and different diamines, prepared from  $\text{SnX}_2$  as a starting material and characterised by physicochemical and spectroscopic data.

### EXPERIMENTAL

Anhydrous tin(II) bromide was prepared by literature method<sup>12</sup>. The aromatic compound (2 mmol) and the diamine (1 mmol) was mixed in methanol and refluxed for 3 hrs for symmetrical Schiff bases (for unsymmetrical Schiff bases one aromatic compound (1 mmol), diamine (1 mmol) and other aromatic compound (1 mmol)). TLC suggested complete conversion of the starting materials to the Schiff base. Then tin compound  $\text{SnX}_2$  (1 mmol) was added and the mixture was refluxed again for 3 hrs. The resulting solid product was washed with methanol and air-dried.

The microanalyses of these complexes were carried out at CDRI, Lucknow. Conductance measurements in DMF were made at room temperature using a Digison Electronics Conductivity Bridge. Infrared spectra of ligands and the complexes were recorded in nujol and KBr respectively on Perkin-Elmer 457 spectrometer.

The X-ray photoelectron spectra were recorded on a VG Scientific ESCA-3 MK II electron spectrometer. The  $\text{MgK}_\alpha$  X-ray line (1253.6 eV) was used for photoexcitation. The  $\text{Cu}2p_{3/2}$  (BE =  $932.8 \pm 0.2$ ) and  $\text{Au}4f_{7/2}$  (BE =  $83.8 \pm 0.1$ )

lines were used to calibrate the instrument and Ag3d<sub>5/2</sub> (BE = 368.2) was used for cross-checking<sup>7</sup>. All the spectra were recorded using the same spectrometer parameter of 50 eV pass energy and 4 mm slit width. The reduced full width at half maximum (FWHM) at Au4f<sub>7/2</sub> (BE = 83.8 eV) level under these conditions was 1.2 eV.

The powdered sample was mixed with high purity silver powder to reduce the charging effect. A thin layer of such a sample was pressed on a gold metal gauze which was welded to a nickel sample holder. The Ag3d<sub>5/2</sub> level (BE = 368.2 eV) obtained from this sample was sharp and did not show any observable shift. Thus the charging of the sample if at all present was negligible<sup>8</sup>. The spectra were recorded in triplicate in the region of interest. In most of the cases the binding energies were reproducible within  $\pm 0.1$  eV. The usual leastsquares fitting procedure of determining peak position, line width and area was used.

## RESULTS AND DISCUSSION

Equimolar addition compounds of SnX<sub>2</sub> (X = Br<sup>-</sup> or Cl<sup>-</sup>) with the following aromatic Schiff base ligands have been synthesised.

SB<sub>1</sub> = bis-benzaldehyde-*o*-phenylenediamine

SB<sub>2</sub> = bis-acetophenone-*o*-phenylenediamine

SB<sub>3</sub> = bis-cinnamaldehyde-*o*-phenylenediamine

SB<sub>4</sub> = bis-acetophenone-ethylenediamine

SB<sub>5</sub> = bis-benzaldehyde-ethylenediamine

SB<sub>6</sub> = bis-cinnamaldehyde-ethylenediamine

SB<sub>7</sub> = bis-benzaldehyde-1,3-diaminopropane

SB<sub>8</sub> = bis-acetophenone-1,3-diaminopropane

SB<sub>9</sub> = bis-cinnamaldehyde-1,3-diaminopropane

SB<sub>10</sub> = acetophenone-benzaldehyde ethylenediamine

SB<sub>11</sub> = acetophenone-cinnamaldehyde ethylenediamine

SB<sub>12</sub> = acetophenone-2-naphthaldehyde ethylenediamine

SB<sub>13</sub> = benzaldehyde-cinnamaldehyde ethylenediamine

SB<sub>14</sub> = benzaldehyde-2-naphthaldehyde ethylenediamine

SB<sub>15</sub> = cinnamaldehyde-2-naphthaldehyde ethylenediamine

SB<sub>16</sub> = bis-2-naphthaldehyde ethylenediamine

All these complexes are stable with high melting points. They all are insoluble in common organic solvents except DMF and DMSO. Microanalytical data suggest that these complexes have the composition of SnX<sub>2</sub>·SB. The low conductance values reveal the non-electrolytic nature of these complexes.<sup>9</sup>

The bands observed in the region of 1610–1670 cm<sup>-1</sup> in the IR spectra of all the ligands and complexes are assigned to  $\nu_{C=N}$  vibration<sup>10</sup>. The ligand  $\nu_{C=N}$

bands around  $1610\text{ cm}^{-1}$  in the ligands shifted to  $1635\text{--}1670\text{ cm}^{-1}$  in the complexes due to an increase of bond order on coordination<sup>11</sup>. The bands at *ca.*  $305\text{--}330\text{ cm}^{-1}$  and *ca.*  $280\text{--}285\text{ cm}^{-1}$  in the complexes may be assigned to  $\nu\text{Sn-N}$  and  $\nu\text{Sn-Cl}$  respectively<sup>3</sup>. The  $\nu\text{Sn-Br}$  frequency could not be identified in the present investigation as it lies beyond the recorded range of vibration.

The binding energy data of  $\text{Sn}3p_{1/2, 3/2}$ , N1s and Xnp photoelectron peaks for the  $\text{SnX}_2$ ,  $\text{SnX}_2\text{-SB}$  (X = Br, Cl) complexes showed that  $\text{Sn}3p_{1/2, 3/2}$  binding energy is higher in  $\text{SnCl}_2$  than  $\text{SnBr}_2$ . It may be further noted that  $\text{Sn}3p$ -BE value in the starting material  $\text{SnX}_2$  is higher than in (having same X)  $\text{SnX}_2\text{-SB}$  complexes. One can conclude from these observations that the Schiff base ligands are coordinated to tin metal ion<sup>8</sup>. Furthermore, the N1s photoelectronpeak in each complex has shown single symmetrical nature, suggested both nitrogen atoms are coordinated to the tin metal ion.<sup>8</sup>

## REFERENCES

1. E.G. Rochow and E.W. Abel, *The Chemistry of Germanium, Tin and Lead*, Pergamon Texts in Inorganic Chemistry, **14**, 43 (1973)
2. B.K. Dwivedi and T.N. Srivastava, *J. Indian Chem. Soc.*, **12**, 246 (1985).
3. B.K. Dwivedi, Kiran Bhatnagar and A. K. Srivastava, *Synth. React. Inorg. Met-Org. Chem.*, **16**, 841 (1986).
4. \_\_\_\_\_, *Indian J. Chem.*, **26**, 618 (1987).
5. \_\_\_\_\_, *Synth. React. Inorg. Met.-Org. Chem.*, **16**, 715 (1986).
6. Anil Varshney and J.P. Tandon, *Proc. Indian Acad. Sci.*, **93**, 1259 (1984).
7. S. Evans, *Handbook of X-ray and Ultra-violet Photoelectron Spectroscopy* (ed. D. Briggs), Hayden, Rochelle Park, NJ 128 (1975).
8. Shekhar Srivastava, *Applied Spectros. Reviews*, **22**, 401 (1986).
9. W.J. Greary, *Coord. Chem. Rev.*, **7**, 81 (1971).
10. J.R. Thornback and G. Wilkinson, *J. Chem. Soc., Dalton Trans.*, **2**, 110 (1978).
11. D.H. Bush and J.C. Bailor (Jr.), *J. Am. Chem. Soc.*, **78**, 1137 (1956).
12. G.W.A. Fowles and I.A. Khan, *J. Less Comm. Metals*, **15**, 209 (1968).

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