

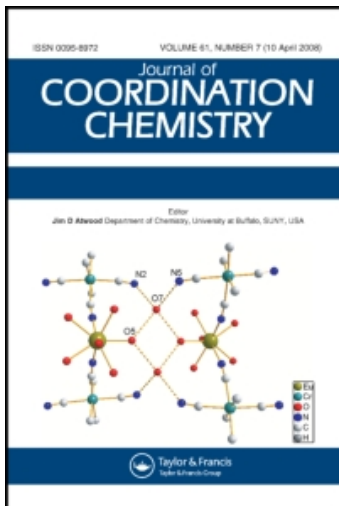
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SYNTHESES AND CHARACTERIZATION OF NEW ADDITION COMPLEXES OF TIN DICHLORIDE WITH AROMATIC SCHIFF BASES

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SYNTHESES AND CHARACTERIZATION OF NEW ADDITION COMPLEXES OF TIN DICHLORIDE WITH AROMATIC SCHIFF BASES

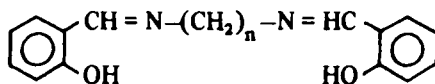
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New addition complexes of tin dichloride with quadridentate Schiff bases have been obtained. Elemental analysis, IR and MS studies indicate the presence of 1:2 (ligand/SnCl₂) adducts, which have an ionic structure.

INTRODUCTION

Addition complexes of transition metals with Schiff bases have attracted much attention in the past.¹⁻⁷ The stereochemistry of such complexes has been the subject of studies both in solution and in the solid state. In the present work, in an attempt to synthesize tetracoordinate complexes of tin(II) with the three aromatic Schiff bases(I)-(III) starting from tin dichloride in methanol solution, we have obtained new 1:2 (ligand: tin dichloride) Sn(II) addition complexes.



(I) $n = 2$; (II) $n = 4$; (III) $n = 6$.

EXPERIMENTAL

The quadridentate Schiff bases were obtained by direct reaction between salicylaldehyde and the appropriate diamine.¹ The adducts (IV)-(VI) (Scheme 1) described in this paper were prepared according to the following procedure. To a solution of the Schiff base in anhydrous methanol was added dropwise a stoichiometric amount of tin dichloride in anhydrous methanol under a nitrogen atmosphere. The solution was allowed to react at reflux temperatures for 30 minutes.

The precipitation of the addition complexes is immediate for compounds (V) and (VI), while in the case of the adduct (IV), the precipitation occurs after some minutes. After filtration of the hot solution, the precipitates were dried under reduced pressure at room temperature. Attempts to obtain the Sn(II) complexes by carrying out the reaction in the presence of base have failed. The melting points and elemental analyses of the adducts are reported in Table I.

By evaporating the filtrate it was possible to recover a 45-50% yield of unreacted schiff base. The infrared spectra were determined with a Perkin-Elmer 684 spectrometer (in the range 4000-200 cm⁻¹) in KBr pellets. Mass spectra were obtained using quartz probes placed in the direct insertion inlet of an electron impact (70 eV) LKB-9000 S mass spectrometer. Conductance measurements were carried out with a Wayne-Kerr bridge.

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TABLE I
Analytical data for the adducts

Compound	M.P. (°)	Analysis ^a				
		C	H	N	Cl	Sn
(IV)	270-272	28.54 (29.07)	2.13 (2.49)	3.99 (4.32)	20.12 (21.9)	35.84 (36.66)
(V)	220-222	32.60 (32.00)	3.05 (2.98)	4.21 (4.14)	21.12 (20.99)	34.99 (35.14)
(VI)	198	34.76 (34.14)	3.49 (3.43)	4.05 (3.98)	21.02 (20.15)	32.84 (33.73)

^aCalculated figures are given in parentheses.

RESULTS AND DISCUSSION

The adducts described in this work are yellow solids, exhibit no crystallinity, and are insoluble in common organic solvents. They are air-sensitive and show also a marked thermal instability (cannot be sublimed under reduced pressure). The elemental analysis are in accordance with complexes of stoichiometry Sn_2LCl_4 and such a composition is confirmed by the fact that after the precipitation of the adducts is complete, the unreacted ligands can be recovered in 50% yield by evaporating the solvent.

In the IR spectra of the ligands as well as their adducts, no bands are observed in the region $3500\text{--}3700\text{ cm}^{-1}$, attributable to the stretching vibration of the free hydroxyl group, indicating that the ring formed by an intramolecular hydrogen bond in the ligands is retained in the adducts.³

In compounds in which all the oxygen atoms are bonded, the infrared spectra show only a single band in the $1500\text{--}1560\text{ cm}^{-1}$ region,^{1,4} and this band is at about 1550 cm^{-1} . The bands occurring at $1650\text{--}1660\text{ cm}^{-1}$ and $1540\text{--}1550\text{ cm}^{-1}$ attributed to $\nu_{\text{C}=\text{N}}$ and $\nu_{\text{C}=\text{O}}$, respectively, appear to be shifted with respect to corresponding free ligand absorptions, in line with *N* and *O* to Sn coordination.⁵

TABLE II
Mass spectral data for the adducts at 180°.

Compound (IV)	Compound(V)	Compound (VI)
385 (100)	414 (10)	441 (65)
266 (76)	378 (2)	378 (27)
253 (31)	350 (5)	335 (22)
238 (57)	294 (2)	322 (43)
225 (45)	266 (5)	308 (39)
212 (12)	238 (7)	266 (57)
193 (10)	225 (3)	253 (41)
155 (10)	174 (25)	225 (42)
132 (11)	120 (5)	212 (18)
120 (12)	36 (100)	120 (31)
36 (25)		36 (100)

^aData are for *m/z* with relative intensities in parentheses.

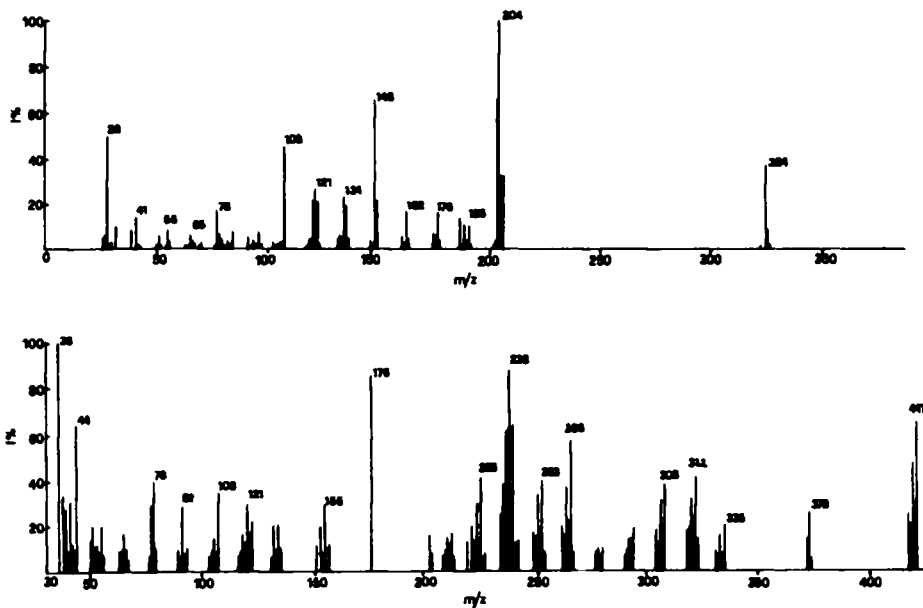
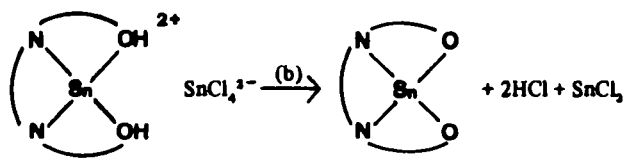
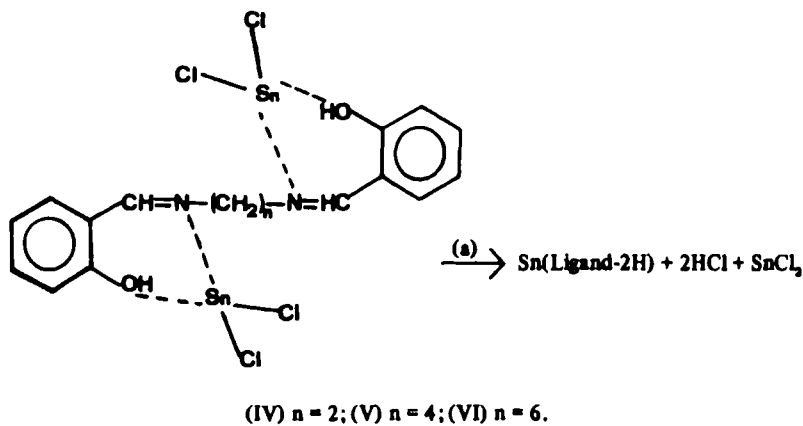


FIGURE 1 (a) Mass spectrum (70 eV) of the ligand (III) at 180° and (b) mass spectrum (70 eV) of the adduct (VI) at the same temperature.

The electron impact 70 eV mass spectra were collected at different temperatures in order to obtain information on the chemical species present in the vapor phase of the adducts and related ligands. The mass spectral data for the compounds (IV)-(V) are reported in Table II.

SCHEME 1



In Figure 1 is reported, as an example, the mass spectra at 180° (maximum ionic current) for adduct (VI) and the related ligand (III) (the fragmentations of the other adducts and ligands were comparable). The spectra at lower temperatures showed neither different fragments nor marked changes in the relative intensities of the fragments. An EI fragment appears at m/z 441 with evolution of SnCl_2 (which is not detected) and HCl (m/z 36). Furthermore, EI fragments of aromatic derivatives containing tin (m/z 378, 335, 308, 266, 253, 238, 225, 212) and fragments derived from the ligand can be detected.

Two different possible hypotheses for the structure of the adducts may be put forward. In the first each ligand is coordinated to two SnCl_2 molecules as shown in Scheme 1(a), and in the second the complexes are of the type $[\text{M}(\text{ligand})] [\text{MCl}_4]$ as shown in Scheme 1(b). In order to differentiate between the two structures we have carried out conductance experiments in anhydrous DMF. The values of Λ_m (1×10^{-4} M) in DMF are 86, 89 and 90 $\text{ohm}^{-1} \text{cm}^2 \text{mol}^{-1}$ for the adducts (IV), (V) and (VI), respectively. These values are very close to those observed for similar ionic complexes.⁷ In addition the Λ_m (1×10^{-4} M) values in DMF for ionic Et_4NBr and ligand (I) chosen as references were found to be 70 and 3 $\text{ohm}^{-1} \text{cm}^2 \text{mol}^{-1}$, respectively.

On the basis of the conductance results we thus conclude that the ionic structure $[\text{M}(\text{ligand})] [\text{MCl}_4]$ is correct.

REFERENCES

1. D.P. Graddon and D.G. Weeden, *Aust. J. Chem.*, **16**, 980 (1963); **17**, 607 (1964); D.P. Graddon, K.B. Heng and E.C. Watton, *ibid.*, **19**, 1801 (1966); G.E. Batley and D.P. Graddon, *ibid.*, **20**, 877 (1967); *ibid.*, **20**, 885 (1967).
2. M. Kato, K. Imai, Y. Muto, T. Tokii and H.B.J. Jonassen, *J. Inorg. Nucl. Chem.*, **35**, 109 (1973).
3. F.A. Bottino, E. Libertini, O. Puglisi and A. Recca, *ibid.*, **41**, 1725 (1979).
4. R. Barbieri, G. Alonzo, A. Silvestri, N. Burriesci, N. Bertazzi, G. Stocco and E. Pellerito, *Gazz. Chim. Ital.*, **104**, 885 (1974).
5. R. Barbieri and R.H. Herber, *J. Organomet. Chem.*, **42**, 65 (1972).
6. K. Kawakami, M. Miya-Uchi and T. Tanaka, *J. Inorg. Nucl. Chem.*, **33**, 3773 (1971).
7. A. Van den Berghen, R.J. Cozens and K.S. Murray, *J. Chem. Soc. A.*, 3060 (1970).