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PREPARATION AND CHARACTERISATION OF $[SnX_2.L^{1-10}]$ COMPLEXES WITH MACROCYCLIC LIGANDS

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ABSTRACT

Thirty complexes of the type $[SnX_2.L^{1-10}]$ (where X = Cl or Br or CH_3COO and L^{1-10}= macrocyclic ligands derived from squaric acid and different aliphatic diamines) are synthesized and characterized by elemental analysis, molar conductivity, Infra-red and XPS data. A distorted octahedral geometry is also established.

INTRODUCTION

In the continuation of synthesis of $[SnX_2.L^{1-10}]$ (where X = Cl or Br or CH_3COO and L^{1-10}= macrocyclic Schiff's base ligands derived from condensation of trimestic acid and p-phathalic acid with various aliphatic diamines [1-2]; in this research paper the synthesis and characterization of $[SnX_2.L^{1-10}]$ molecular adducts (where X = Cl or Br or CH_3COO and L^{1-10} = macrocyclic Schiff's base ligands derived from condensation of squaric acid i.e. 3,4-dihydroxy-3-cyclobutene-1-2-dione with different aliphatic diamines i.e.

L^1= $NH_2CH_2CH_2CH_2NH_2$
L^2= $NH_2CH_2CH(CH_3)CH_2NH_2$
L^3= $NH_2CH_2NH(CH_2)_2NH_2$
L^4= $NH_2CH_2C(CH_3)_2CH_2NH_2$
L^5= $NH_2(CH_2)_6NH_2$
L^6= $NH_2(CH_2)_7NH_2$
L^7= $NH_2(CH_2)_8NH_2$
L^8= $NH_2(CH_2)_9NH_2$
L^9= $NH_2(CH_2)_{10}NH_2$ and
L^{10}= $NH_2(CH_2)_{10}NH_2$

will be performed and their structure and geometry will be proposed on basis of physicochemical methods.

KEYWORDS

Elemental analysis, Molar conductivity, Macrocyclic ligand, IR and X-ray Photoelectron spectra.

MATERIAL AND METHODS

On melting point apparatus melting points were determined. CDRI, Lucknow, (India) carried out C, H, N and Cl. At room temperature in acetone molar conductance were measured on conductivity bridge. On Infra-red Perkin–Elmer spectrometer infra-red spectra were recorded using CsI Pellets. On VG scientific ESCA-II spectrometer X-ray photoelectron spectra were recorded using MgKα as X-ray source.

PREPARATION OF [SnX$_2$.L$^{1-10}$] COMPLEXES

Squaric acid or 3-4-dihydroxy-3-cyclobutene-1-2-dione (2mmol) in 50 ml dry methanol was mixed with different aliphatic diamines (2mmol) i,e.

$NH_2CH_2CH_2CH_2NH_2$ or $NH_2CH_2CH(CH_3)CH_2NH_2$ or $NH_2CH_2NH(CH_2)_2NH_2$ or $NH_2CH_2C(CH_3)_2CH_2NH_2$ or $NH_2(CH_2)_6NH_2$ or $NH_2(CH_2)_7NH_2$ or $NH_2(CH_2)_8NH_2$ or $NH_2(CH_2)_9NH_2$ or $NH_2(CH_2)_{10}NH_2$ or $NH_2(CH_2)_{12}NH_2$

and then refluxed for three hours and then added SnX_2 (1mmol) (where X = Cl or Br or CH_3COO) solution into above refluxing flask and refluxed for another three hours. The yellow precipitate was obtained, filtered and recrystallised by benzene: pet ether 9:l and air-dried. (Fig.1).

RESULTS AND DISCUSSION

The interaction of squaric acid i.e. 3-4-dihydroxy-3-cyclobutene-1-2-dione with different aliphatic diamines in 2:2 molar ratio resulted ten macrocyclic Schiff's base ligands i.e. L^1 or L^2 or L^3 or L^4 or L^5 or L^6 or L^7 or L^8 or L^9 or L^{10} (Fig 1). Further, when SnX_2 (1m mol)) (where X = Cl or Br or CH_3COO) was added in each prepared ligand and refluxed again, a yellow precipitate was obtained.

All the precipitate were yellow solids and stable. The color elemental analysis for C, H, N and Cl were observed within ±0.5%. The molar conductance of each these molecular adducts were found below 40 ohm^{-1} cm^2 mol^{-1} in acetone at room temperature, suggested non-electrolyte [3] The fundamental infra-red frequencies for $\nu_{Sn\text{-}N}$ and $\nu_{Sn\text{-}Cl}$ in these molecular adducts were observed in the range of 436-480 cm^{-1} and 485-496 cm^{-1} respectively [4-5].

The Sn3p1/2,3/2 and N1s photoelectron peaks binding energies (eV) for ligands, SnX_2 and [SnX_2.L^{1-10}] are listed in Table I. The Sn3p1/2,3/2 photoelectron peaks binding energies (eV) in SnX_2 and [SnX_2.L^{1-10}]; it was observed that Sn3p1/2,3/2 photoelectron peaks binding energies (eV) are more in SnX_2 than [SnX_2.L^{1-10}]. One can conclude from these observations that electron density is more in [SnX_2.L^{1-10}] than SnX_2 due to coordination ligand (Table 1 & Fig. 2). Furthermore, comparative observations of N1s photoelectron peaks binding energies (eV) in each ligand with their each [SnX_2.L^{1-10}] molecular adducts have shown two N1s photoelectron peaks in [SnX_2.L^{1-10}] in 1:3 intensity ratio, one N1s photoelectron peak with high intensity was observed at high binding energy side than N1s photoelectron peak of each ligand, while one N1s photoelectron peak with low intensity was observed at the same position as in the ligand N1s photoelectron peak. One can conclude from these observations that out of four nitrogen atoms in ligand only three are coordinated to Tin(II) metal ion and one nitrogen atom is uncoordinated [6] (Table 1 & Fig. 3). Furthermore, Sn3s Photoelectron peak in each [SnX_2.L^{1-10}] molecular adduct have shown a single symmetrical peak i.e. multiple splitting is not observed, suggested each [SnX_2.L^{1-10}] molecular adduct are diamagnetic [6].

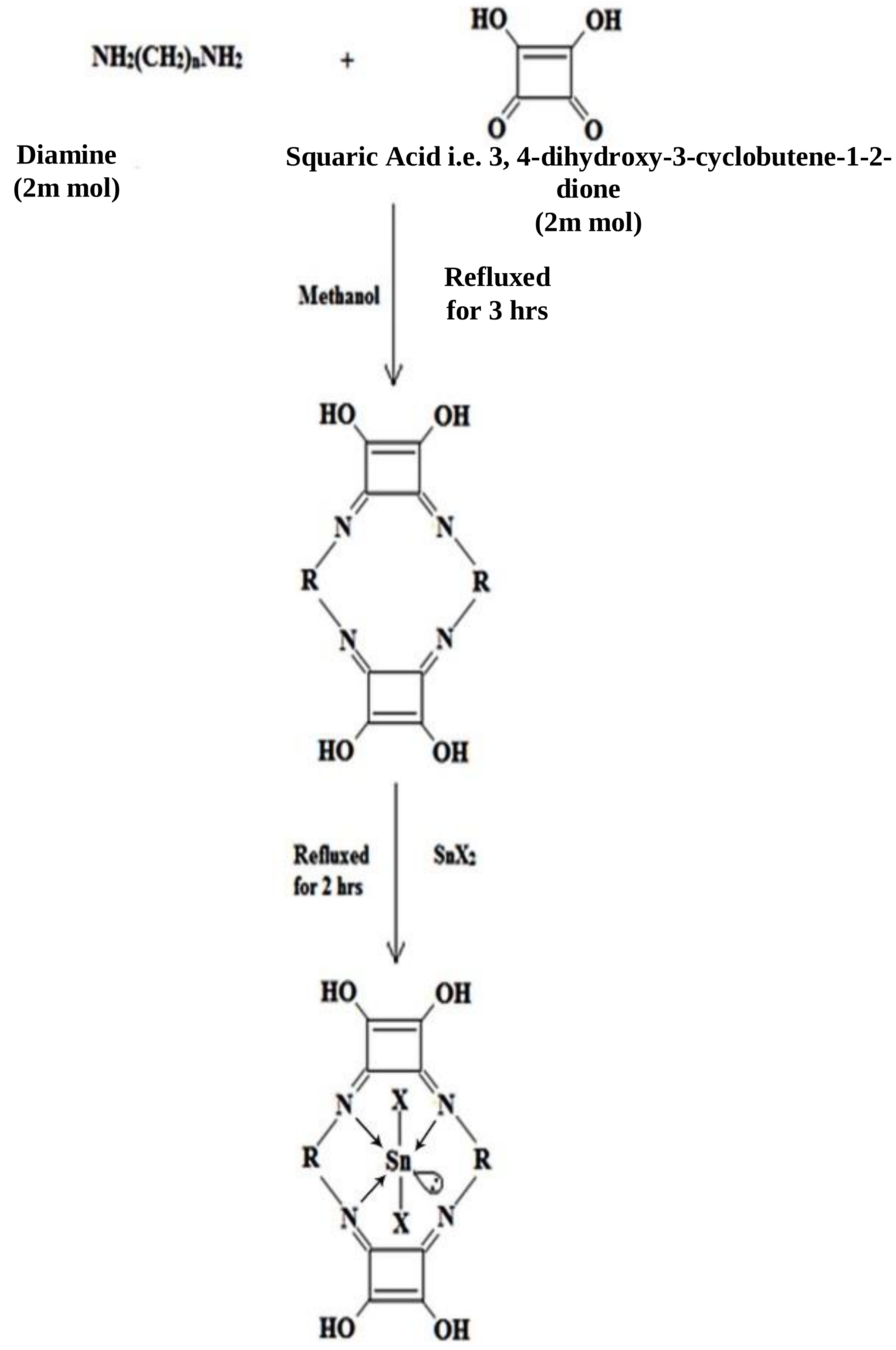
Table 1

$Sn3p_{1/2,3/2}$ and N1s binding energies (eV) in ligand, SnX_2 and [SnX_2.L^{1-10}]

S. No.	Ligand and Compound	$Sn3p_{1/2,3/2}$		N1s	
		$Sn3p_{1/2}$	$Sn3p_{3/2,}$	Uncoord.	Coord.
1	L^1	-	-	400.2	-
2	L^2	-	-	400.2	-

3	L^3	-	-	400.2	-
4	L^4	-	-	400.2	-
5	L^5	-	-	400.2	-
6	L^6	-	-	400.2	-
7	L^7	-	-	400.2	-
8	L^8	-	-	400.2	-
9	L^9	-	-	400.2	-
10	L^{10}	-	-	400.2	-
11	$SnCl_2$	758.8	516.8	-	-
12	$SnCl_2.L^1$	757.2	515.2	400.2	404.8
13	$SnCl_2.L^2$	757.2	515.2	400.2	404.8
14	$SnCl_2.L^3$	757.2	515.2	400.2	404.8
15	$SnCl_2.L^4$	757.2	515.2	400.2	404.8
16	$SnCl_2.L^5$	757.2	515.2	400.2	404.8
17	$SnCl_2.L^6$	757.2	515.2	400.2	404.8
18	$SnCl_2.L^7$	757.2	515.2	400.2	404.8
19	$SnCl_2.L^8$	757.2	515.2	400.2	404.8
20	$SnCl_2.L^9$	757.2	515.2	400.2	404.8
21	$SnCl_2.L^{10}$	757.2	515.2	400.2	404.8
22	$SnBr_2$	758.6	516.6	-	-
23	$[SnBr_2.L^1]$	757.0	515.0	400.2	404.6
24	$[SnBr_2.L^2]$	757.0	515.0	400.2	404.6
25	$[SnBr_2.L^3]$	757.0	515.0	400.2	404.6
26	$[SnBr_2.L^4]$	757.0	515.0	400.2	404.6
27	$[SnBr_2.L^5]$	757.0	515.0	400.2	404.6
28	$[SnBr_2.L^6]$	757.0	515.0	400.2	404.6
29	$[SnBr_2.L^7]$	757.0	515.0	400.2	404.6
30	$[SnBr_2.L^8]$	757.0	515.0	400.2	404.6
31	$[SnBr_2.L^9]$	757.0	515.0	400.2	404.6
32	$[SnBr_2.L^{10}]$	757.0	515.0	400.2	404.6
33	$Sn(CH_3COO)_2$	758.2	516.2	-	-
34	$[Sn(CH_3COO)_2.L^1]$	757.0	515.0	400.2	404.8
35	$[Sn(CH_3COO)_2.L^2]$	757.0	515.0	400.2	404.8
36	$[Sn(CH_3COO)_2.L^3]$	757.0	515.0	400.2	404.8
37	$[Sn(CH_3COO)_2.L^4]$	757.0	515.0	400.2	404.8
38	$[Sn(CH_3COO)_2.L^5]$	757.0	515.0	400.2	404.8
39	$[Sn(CH_3COO)_2.L^6]$	757.0	515.0	400.2	404.8
40	$[Sn(CH_3COO)_2.L^7]$	757.0	515.0	400.2	404.8
41	$[Sn(CH_3COO)_2.L^8]$	757.0	515.0	400.2	404.8
42	$[Sn(CH_3COO)_2.L^9]$	757.0	515.0	400.2	404.8
43	$[Sn(CH_3COO)_2.L^{10}]$	757.0	515.0	400.2	404.8

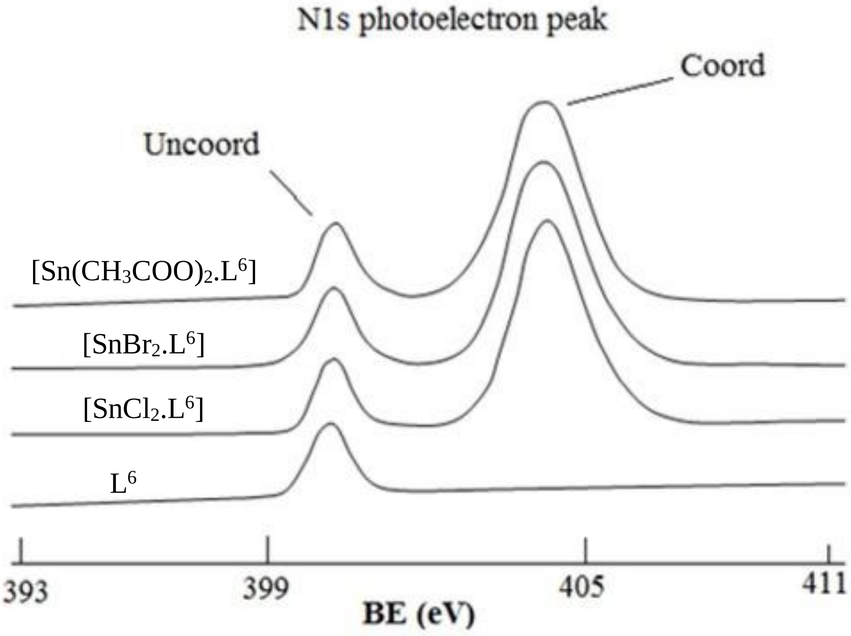
Figure - 1



Preparation of $[SnX_2.L]$ (where X=Cl or Br or CH_3COO and R= $-CH_2CH_2CH_2-$; $-CH_2CH(CH_3)CH_2-$; $-CH_2NH(CH_2)-$; $-CH_2C(CH_3)_2CH_2-$; $-(CH_2)_6-$; $-(CH_2)_7-$; $-(CH_2)_8-$; $-(CH_2)_9-$; $-(CH_2)_{10}-$ and $-(CH_2)_{12}-$)

$Sn3p_{1/2}$ Binding Energies (eV) in $SnCl_2$ & [$SnCl_2L^{1-10}$] Complexes

Figure - 3



N1s Binding Energies (eV) in L^6 & [$SnX_2.L^6$] Complexes

CONCLUSION

On the basis of elemental analysis, molar conductivity, IR and XPS data, the structure of [$SnX_2.L^{1-10}$] may be propose as given in fig 1 and a distorted octahedral geometry may be established.

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