Int. J. Chem. Sci.: 9(3), 2011, 989-995
ISSN 0972-768X
www.sadgurupublications.com

SYNTHESIS OF Sn (II) DIHYDROPHOSPHAZENIDE AND ITS ANTIMICROBIAL STUDIES

ATUL VASHISTHA and S. P. S. JADON*

Department of Chemistry, S. V. College, ALIGARH – 202001 (U.P.) INDIA

ABSTRACT

Complex of Sn (II) was synthesized with hexahydrocyclotriphosphazene and studied with the help of Mass, IR and EPR spectra, assigning its molecular formula as $(N_3P_3H_2)_6$–Sn_3–P_4N_5 with quadridentated geometry. The complex has also found effective against *Klebsiella* gm-ve and *Staphylococcus* gm+ve bacteria.

Key words: Sn (II), Phosphazenide, Mass, IR, EPR.

INTRODUCTION

Various complexes of adducts of $(NPCl_2)_3$ with metals have been reported[1-8], but a few complexes of $(NPH_2)_3$ with metals have been synthesized[9-12]. The investigations of reaction product of HHCPT with Sn (II) chloride are reported herewith.

EXPERIMENTAL

On the reduction of $(NPCl_2)_3$, by Na/EtOH, hexahydrocyclotriphosphazene $(NPH_2)_3$, was prepared, as white mass. Which was separated, washed with dry C_2H_5OH and ether, dried and stored in vacuo. The complex of $(NPH_2)_3$ with $SnCl_2$ was prepared by refluxing both in eqimolar ratio (1 : 1) in DMF for 6 to 8 h.

The yellowish mass, obtained, was separated, washed with C_6H_5Cl, C_2H_5OH & ether, dried and stored in vacuum desiccator over fused $CaCl_2$.

The complex was analysed qualitatively & quantitatively by well known methods[13]. EPR and mass spectra were recorded on Varians X–E–4 band (4 to 8 K Gauss at RT & LNT) & Jeol SX–102 (FAB), sepectrometers respectively. IR spectrum was graphed on Shimadzu 8201 PC (4000 – 400 cm^{-1}) FTIR spectrometer.

*Author for correspondence; E-mail: sps_jadon@yahoo.co.in

For antimicrobial activity of the complex, the in vitro technique was used. The equipments used, were well sterilized in autoclave. The gm-ve bacteria *Klebsiella* and gm+ve bacteria *staphylococcus* were grown by using nutrient broth media incubating at 37^{o}C for 24 h and treated by the complex synthesized.

RESULTS AND DISCUSSION

The quantitative estmations, % found N-23.5, P – 49.7, H – 0.87 and Sn – 25.8 and molecular weight is 1371.5 g/mol, formulated the complex as $(N_3P_3H_2)_6 – Sn_3 – P_4N_5$ which is supported by the prominent mass line at m/z -1370 observed in its mass spectrum (Fig. 1 and Table 1). The other mass lines in its mass spectrum are explained by FAB fragmentation of Sn (II) DHP as follows –

$(N_3P_3H_2)_6 – Sn_3 – P_4N_5$
m/z 1370

- N_3P_2
  m/z 101 (M –3)
- $N_3P_3H_2 – NP_2H_2$
  m/z 217 (M + 2)
- $N_3P_3H_2 – NP_2H_2 – NPH_2$
  m/z 261 (M - 1)
- $N_3P_3H_2 – Sn – N_2P_2H_2$
  m/z 349 (M+2)
- $(N_3P_3H_2)_2 – Sn – NP$
  m/z 437
- $(N_3P_3H_2)_2 – Sn – N_3P_2$
  m/z 497 (M + 1)
- $(N_3P_3H_2)_3 – Sn_2 – N_2$
  m/z 675
- $(N_3P_3H_4)_4 – Sn_2 – N_2P$
  m/z 854 (M+3)
- $(N_3P_3H_2)_6 – Sn_3 – N_2P_2$
  m/z 1265 (M-1)
- $(N_3P_3H_2)_6 – Sn_3 – N_3P_2$
  m/z 1278 (M-2)

Table 1: Mass spectral data of complex, Sn (II) DHP

M/z	Fragments
101	N_3P_2 (M-3)
217	$(N_3P_3H_2) – NP_2H_2$ (M+2)
261	$(N_3P_3H_2) – NP_2H_2 – NPH_2$ (M-1)
305	$(N_3P_3H_2) – Sn – NPH_2$ (M+3)
349	$(N_3P_3H_2) – Sn – N_2P_2H_2$ (M+2)
409	$(N_3P_3H_2)_2 – Sn – N$ (M +3)
437	$(N_3P_3H_2)_2 – Sn – NP$
497	$(N_3P_3H_2)_2 – Sn – N_3P_2$ (M +1)
675	$(N_3P_3H_2)_3 – Sn_2 – N_2$
679	$(N_3P_3H_4)_3 – Sn_2 – N_2$ (M-2)
854	$(N_3P_3H_4)_4 – Sn_2 – N_2P$ (M+3)
925	$(N_3P_3H_2)_4 – Sn_2 – N_3P_3H_4$ (M+2)
1265	$(N_3P_3H_2)_6 – Sn_3 – N_2P_2$ (M-1)
1278	$(N_3P_3H_2)_6 – Sn_3 – N_3P_2$ (M-2)
1370	$(N_3P_3H_2)_6 – Sn_3 – P_4N_5$
1512	$(N_3P_3H_2)_8 – Sn_3 – N_2PH_2$ (M+1)
1655	$(N_3P_3H_2)_8 – Sn_4 – N_2P_2$ (M-3)
1837	$(N_3P_3H_2)_8 – Sn_4 – (P_3N_3)_2$ (M-1)
1936	$(N_3P_3H_4)_8 – Sn_5 – N_2P_2H_4$ (M+1)

The formation of this complex is supported by IR spectrum (Fig. 2), having the frequencies at 601.0 (b), 1021-1353.2 (sextet), 1465.5 (s), 1528.4 – 1547.5 (s), 1636.9 (d), 1704.4 – 1721.5 (tr), 2365.9 (d), 3427.8 (b) cm^{-1}, corresponding to P-N $\rightarrow$ Sn, P-N, H-P-N and P = N bands, suggesting quadridentated co-ordination of P_3N_3 ring to Sn atom.

Both EPR spectra, recorded at RT & LNT have a single peak, indicating paramagnetic character of the complex. Which is also sustained by the values of μ_{eff} = 1.5492 & 2.1155 B. M. & magnetic susceptibility $\chi = 1.003 \times 10^{-3}$ and 2.8550×10^{-3} e.s.u. The value of g_z and g_{av} at LNT are 4.0728 and 2.4429 (Table 2), indicating the sharing of electrons of Sn atom i.e. Sn atom has linked covalently to P-N ring due to pπ-pπ bonding

along with its co-ordination through N atom of P-N rings, because the value of $g_x = g_y$, g_z and g_{av} (at RT, Table 2) are less than 2 corresponding to vacant energy shell of Sn atom to accept the electrons pair from N atom of P-N ring. Thus in the complex pπ-pπ bonding as well as co-ordinate linkage of Sn atom with phosphazene ring persist, forcing to suppose that complex is polymeric in nature & quadridentated, as shown in Fig. 3.

Table 2: EPR spectra of complex

Temp.	Magnetic field H_0. (Gauss)	$g_x = g_y$	g_z	g_{av}	μ_{eff} (B.M.)	$\chi \times 10^{-3}$ esu
	1	**2**	**3**	**4**	**5**	**6**
RT	3431	1.7181	1.9230	1.7890	1.5492	1.0003
LNT	1620	0.8112	4.0728	2.4429	2.1155	2.8550

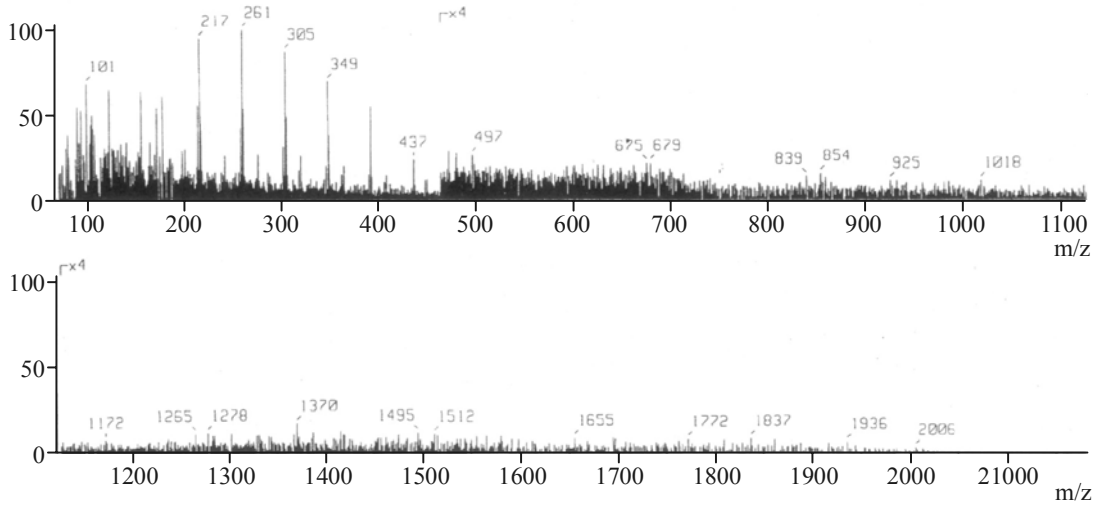

Fig. 1: Mass spectrum of complex

The complex has shown its effectiveness against *Klebsiella* gm-ve and *Staphylococcus* gm+ve bacteria. The inhibition of Sn (II) complex is 14 mm against staphylococcus & 13 mm (Fig. 4) against *Klebsiella* bectraia respectively. *Staphylococcus* bacteria is dangerous to open wounds, it can be responsible for septic. While the urinogential infection in human beings is caused by *Klebsiella* bacteria. The results of antimicrobial studies reveals that the complex of Sn (II) may be used for the treatment of wound healing and urinogential deseases in human beings.

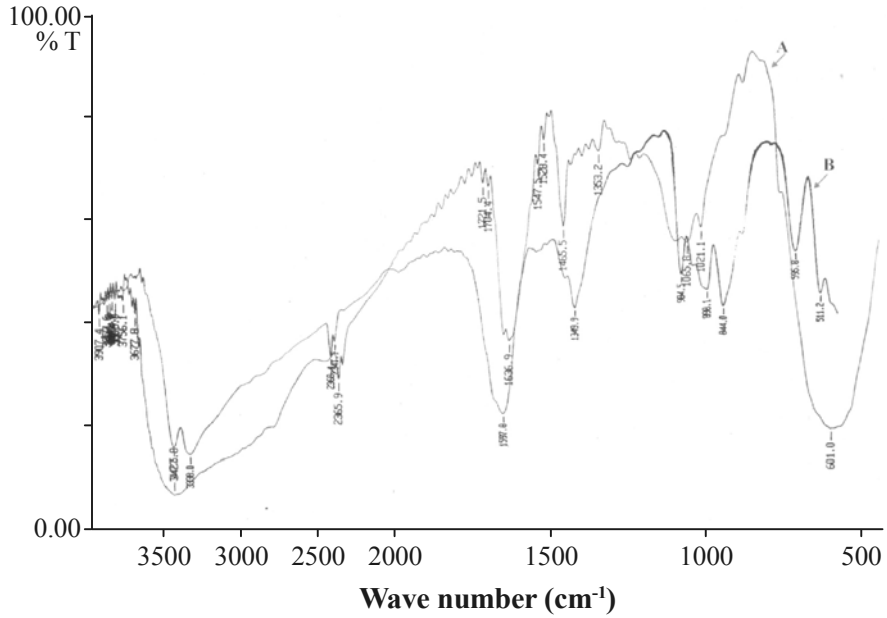

Fig. 2: IR spectrum of complex (A) and Ligand (B)

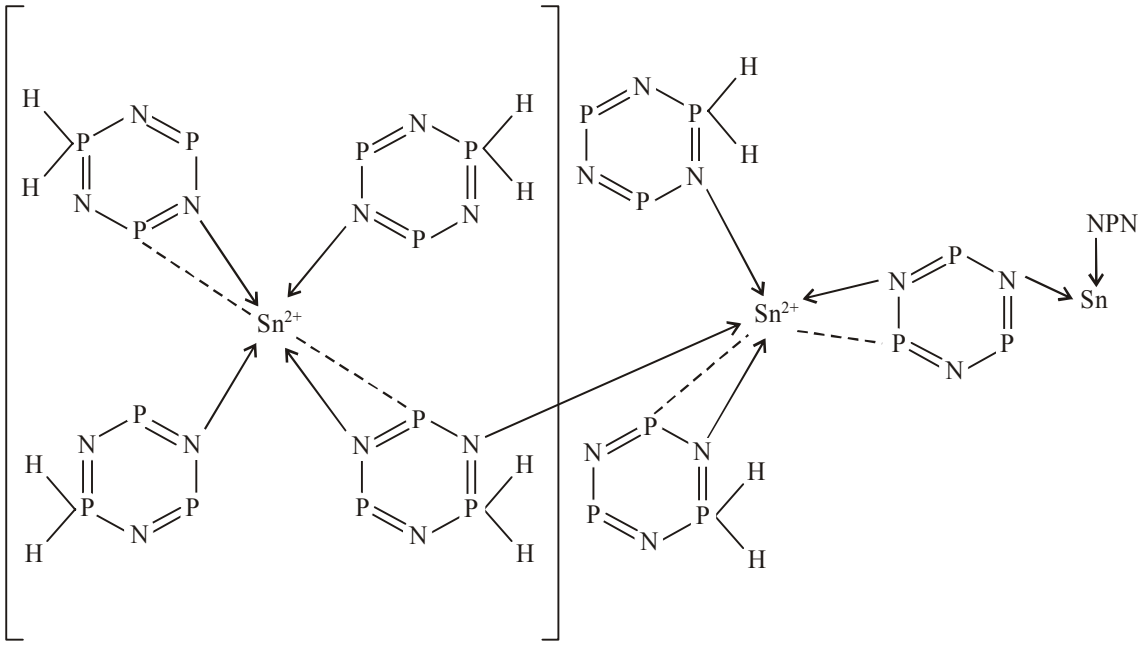

Fig. 3: Structure of complex $(N_3P_3H_2)_6 – Sn_3 – P_4N_5$ Polymer

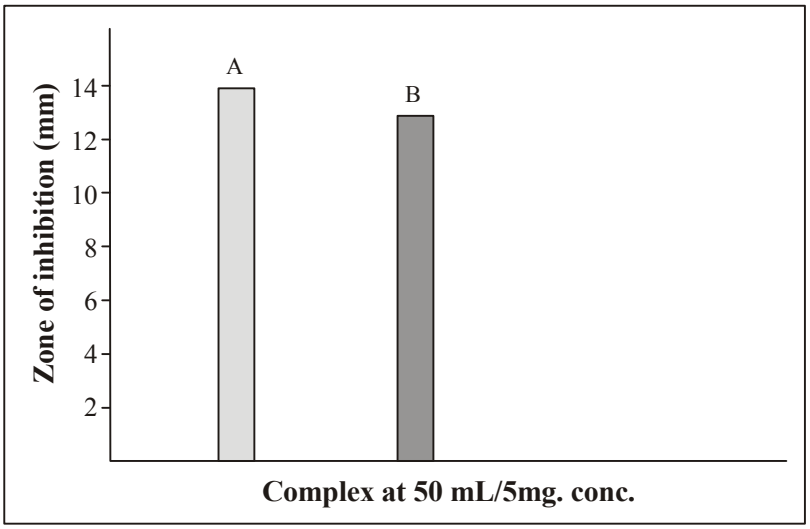

Fig. 4: Bactericidal effect of complex against *Staphylococcus* 'A' and *Klebsiella* 'B'

ACKNOWLEDGEMET

We express our thanks to the Director CDRI, Lucknow for providing the instrumental facility.

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Revised : 31.12.2010 *Accepted : 02.01.2011*