



## **MASS, IR AND EPR SPECTRAL CHARACTERISATION OF Cd (II) PHOSPHAZENIDE**

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### **ABSTRACT**

Complex of Cd (II) with hexahydro-cyclotriphosphazene (HHCTP) was synthesized, and studied with the help of Mass, IR and EPR spectra, assigning its molecular formula as  $(N_3P_3H_4)_5\text{-Cd}$  having pentadentated coordinated linkage and trigonal-bipyramidal geometry.

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

### **INTRODUCTION**

Various complexes and adducts of  $(N\text{P}Cl_2)_3$  with metals have been reported<sup>1-8</sup>, but a few complexes of  $(N\text{P}H_2)_3$  with metals have been synthesized<sup>9-12</sup>. The investigations of reaction product of (HHCTP) with Cd (II) chloride are being reported herewith.

### **EXPERIMENTAL**

$(N\text{P}Cl_2)_3$  was prepared by refluxing  $\text{NH}_4\text{Cl}$  and  $\text{PCl}_5$  in chlorobenzene at the  $150^\circ\text{C}$  for 6 to 12 h by using AnalaR grade chemicals.  $(N\text{P}H_2)_3$  was prepared by the reduction of  $(N\text{P}Cl_2)_3$  with  $\text{Na}/\text{C}_2\text{H}_5\text{OH}$  as reported<sup>13</sup>. The complex of  $(N\text{P}H_2)_3$  with  $\text{CdCl}_2$  was prepared by refluxing both in equimolar ratio (1 : 1) in DMF for 6 to 8 h. White coloured mass obtained was separated, washed with DMF, ethanol and ether, dried and stored in vacuum desiccator over fused  $\text{CaCl}_2$ .

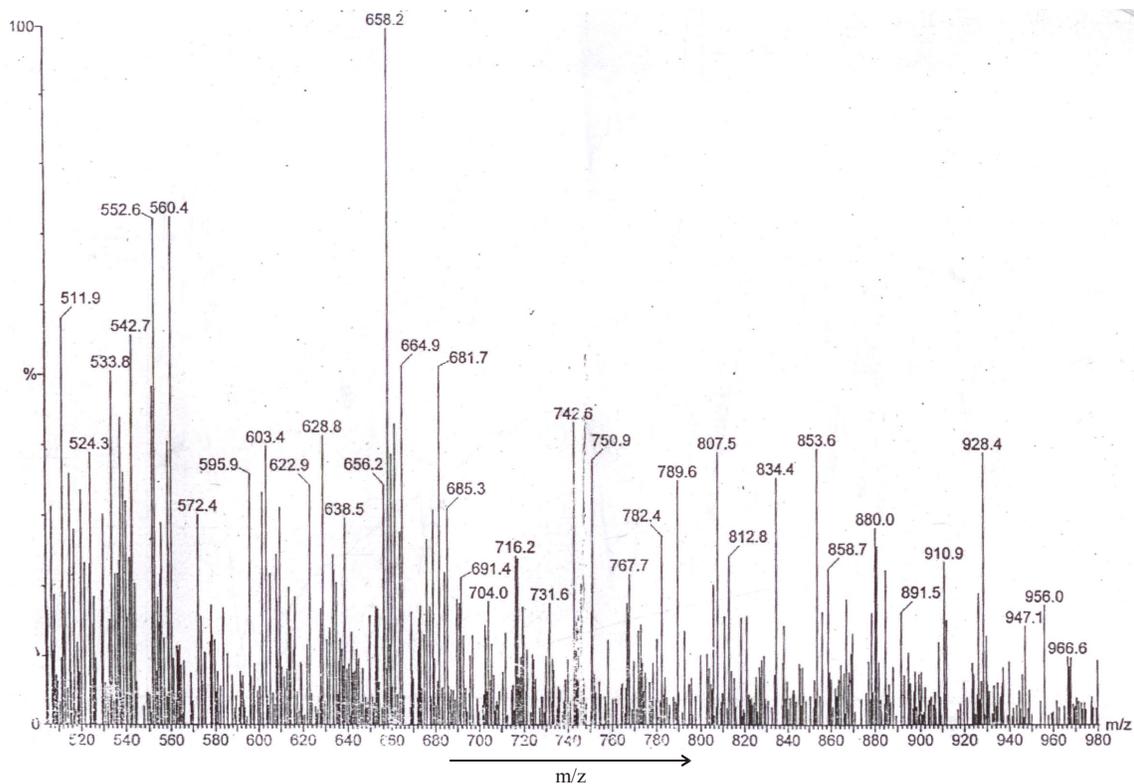
The complex was analysed qualitatively and quantitatively by well known methods<sup>14</sup>. EPR and Mass spectra were recorded subsequently on varians X-E-4 band (4-8K Gauss) and Jeol Sx-102 (FAB) spectrometers. IR spectrum was recorded on Shimadzu 8201, PC ( $4000\text{-}400\text{ cm}^{-1}$ ) FTIR spectrophotometer.

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## RESULT AND DISCUSSION

In the complex, N and P were tested as  $\text{NH}_4^+$  and  $\text{PO}_4^{3-}$  ion and found positive while test for  $\text{Cl}^-$  ion was found negative. The analytical data, % found, N-26.02, P-57.62, H-2.47, Cd-13.87 and molecular weight 807.46 gm/mol formulated the complex as  $(\text{N}_3\text{P}_3\text{H}_4)_5\text{-Cd}$ , which is supported by the prominent mass at  $m/z$  807.5, 767, 782 and 789 appearing in its mass spectrum (Fig. 1) according to the  $(\text{N}_3\text{P}_3\text{H}_4)_5\text{-Cd}$ ,  $(\text{N}_3\text{P}_3\text{H}_2)_4\text{-Cd-N}_3\text{P}_2\text{H}_2$  (M+1),  $(\text{N}_3\text{P}_3\text{H}_2)_4\text{-Cd-N}_2\text{P}_3\text{H}_2$  (M - 1) and  $(\text{N}_3\text{P}_3\text{H}_4)_4\text{-Cd-N}_2\text{P}_3\text{H}_2$  (M - 2), fragments, respectively.



**Fig. 1: Mass spectrum of  $\text{Hg}(\text{N}_3\text{P}_3\text{H}_4)_5\text{-Cd}$**

The other mass lines in its mass pattern may be explained by FAB fragmentation process as –

The formation of this complex is supported by its IR spectrum (Fig. 2) having the vibrations at 555-1380, 1442-1853 and 2340-3778  $\text{cm}^{-1}$ . Subsequently for the H-P-N  $\rightarrow$  M, H-P-N, H-P=N bands, suggesting pentadentated coordinated linkage of  $\text{P}_3\text{N}_3$  ring to  $\text{Cd}^{2+}$  ion.

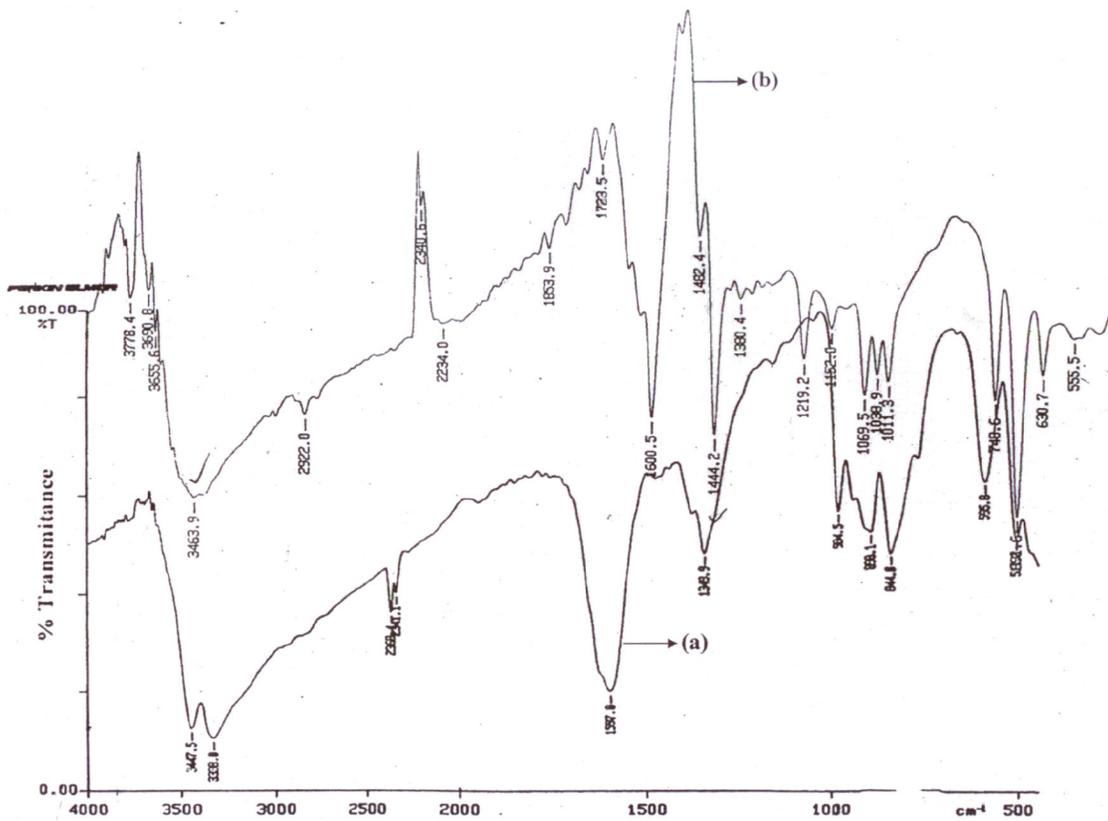
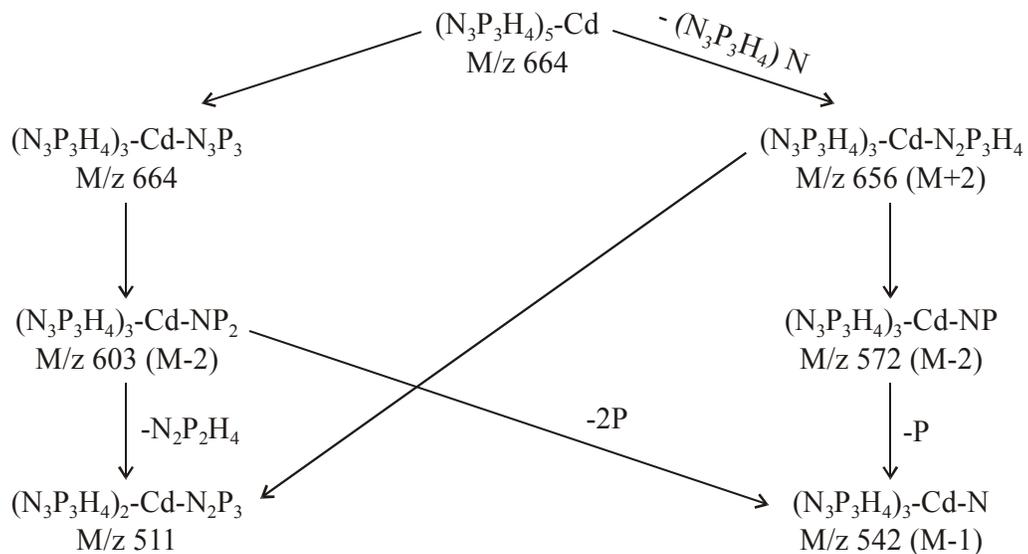
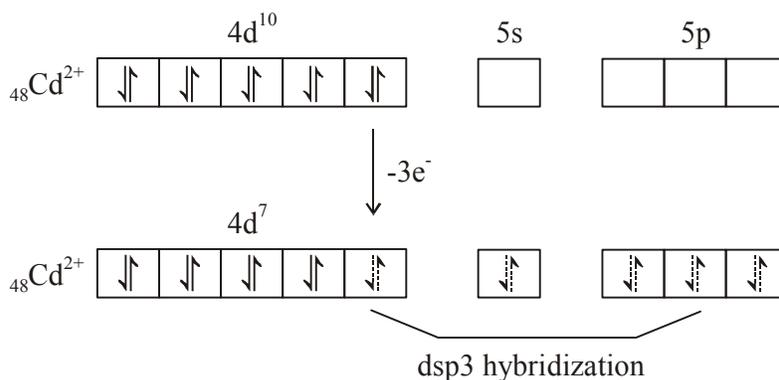


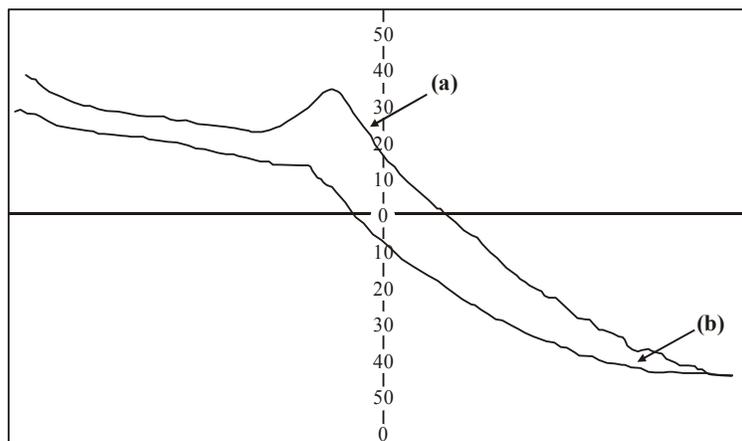
Fig. 2: IR Spectrum of ligand (a) and complex (b)

This view is also supported by the appearance of signals in its EPR spectra at both; RT and LNT (Fig. 3); thus, showing the paramagnetic character of the complex, while the complexes of Cd (II) generally exhibit diamagnetic nature. The paramagnetic character is confirmed by the magnetic momentum  $\mu_{\text{eff}}$  1.1461 B. M. (R. T.), 1.370 B. M. (L. N.T.) and magnetic susceptibility  $\chi_A$   $5.844 \times 10^{-4}$  esu at R. T. and  $1.1973 \times 10^{-3}$  esu at L. N. T. according to one unpaired electron in  $\text{Cd}^{2+}$  ion. The value of  $g = 1.3515 < 2$  indicates the presence of vacant shell in Cd atom to form the coordinate linkage with  $\text{P}_3\text{N}_3$  ring.

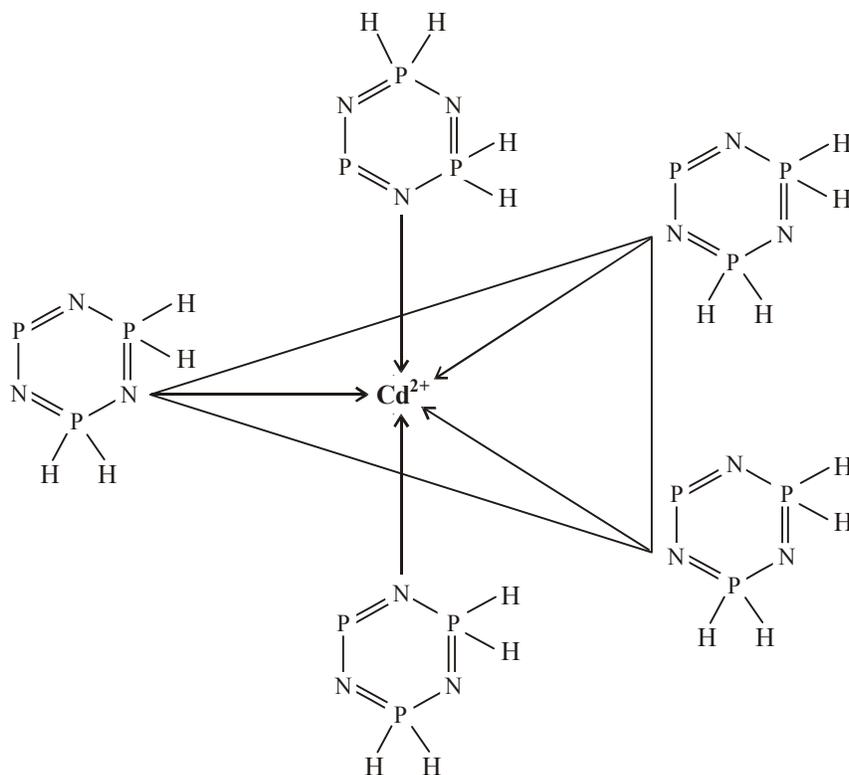
The presence of unpaired electron on  $\text{Cd}^{2+}$  ion may be due to loss of three electrons to reduce phosphorus atoms  $\text{P}(\text{V}) \rightarrow \text{P}(\text{III})$  state of  $\text{N}_3\text{P}_3$  ring as -



Trigonal bipyramidal geometrical complex is formed through  $\text{dsp}^3$  hybridization. The presence of one unpaired electron on  $\text{Cd}^{2+}$  ion expound mono-nuclear complex ruling out any other geometry except trigonal bipyramid<sup>15</sup>. The structure of the complex may be represented as Fig. 4.



**Fig. 3: EPR Spectra of complex at (a) RT and (b) LNT**



**Fig. 4: Structure of  $(N_3P_3H_4)_5\text{-Cd}$**

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