



## **SYNTHESIS AND CHARACTERIZATION OF AMINO ACID SUBSTITUTED CYCLOTRIPHOSPHAZENIDES**

**SANTOSH KUMAR SINGH and S. P. S. JADON\***

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

### **ABSTRACT**

The amino acid, lysine, substituted compound of  $(\text{NPCl}_2)_3$  was synthesized. The complex was studied with the help of Mass, FT-IR and microanalysis assigning its molecular formula as  $(\text{PN})_3[\text{OOC}(\text{CH}_2)_5\text{NH}_2]_6$

**Key words:** Phosphozenides, Amino acid, Lysine.

### **INTRODUCTION**

Due to N atom  $(\text{NPCl}_2)_3$  molecule has much coordinating property and hence, it has formed a large number of complexes with metals<sup>1-6</sup>. On the other hand, there is six symmetric chlorine atoms, which perform substitution reaction with covalent compounds<sup>7</sup>. These substituted and adduct derivatives of  $(\text{NPCl}_2)_3$  have industrial, pharmaceutical and biological importance such as, the complexes of  $(\text{NPCl}_2)_3$  with Cu, Fe and Co were found bactericidal<sup>8-10</sup>.

Adducts of  $(\text{NPCl}_2)_3$  with phenoxy substituted compound are hydraulic lubricant<sup>11</sup>. Polyorganophosphazenes have fire proofing agents<sup>12</sup>, plastics<sup>13</sup> and biological properties,<sup>14</sup> while polymethoxy, ethoxy, amino and aryl substituted polyphosphazene are bioactive<sup>15</sup>. Pt (II) complex of  $(\text{NPCl}_2)_3$  has antitumor activities<sup>16</sup>. Polyaminophosphazenes were found good germicides<sup>17-20</sup>. Water soluble cyclotriphos-phazenes (diamine) Pt (II) conjugated drugs were also found to have antitumor activity<sup>21</sup>.

### **EXPERIMENTAL**

#### **Methodology**

**Chemicals:** Phosphorous pentachloride, amino acids (Lysine), ammonium chloride, chloro-

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\* Author for correspondence; E-mail: sps\_jadon@yahoo.co.in

benzene, ether, alcohol, etc. doubly distilled were used.

### Preparation of hexachlorocyclotriphosphazene

( $\text{NPCl}_2$ )<sub>3</sub> was prepared by the refluxing an equimolar mixture of  $\text{PCl}_5$  and  $\text{NH}_4\text{Cl}$  in chlorobenzene at 140-160°C for 7-8 hours. The unreacted  $\text{NH}_4\text{Cl}$  was removed by filtration and ( $\text{NPCl}_2$ )<sub>3</sub> was obtained, after distillation under reduced pressure.

### Preparation of amino acid (Lysine) substituted phosphazenides

The compound of lysine with ( $\text{NPCl}_2$ )<sub>3</sub> was prepared by the refluxing a mixture of ( $\text{NPCl}_2$ )<sub>3</sub> and lysine (100 mg of each in 1 : 1) at 150-165°C for 7-9 hours using  $\text{C}_6\text{H}_5\text{Cl}$  as a solvent. A brownish precipitate was obtained, which was filtered, washed with chlorobenzene, ether and alcohol. Dried product was stored in vacuum desiccator over fused  $\text{CaCl}_2$ .

### Instrumental studies

A Perkin-Elmer FT-IR spectrophotometer was used to record the IR spectrum (4000-500  $\text{cm}^{-1}$ ). The DART mass spectrum was recorded on a JEOL-Accutof JMS-T100lc Mass spectrometer having a DART source using helium lamp at 350°C. Microanalysis for constituent elements was carried out from CDRI, Lucknow. Molecular weight was determined by Rast's method.

### Observation

The newly synthesized compound was brownish in color, solid, soluble in water and having melting point 350-360°C.

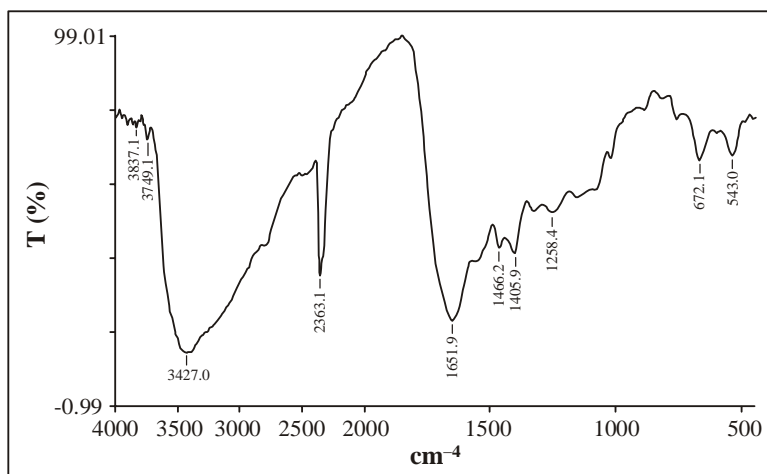
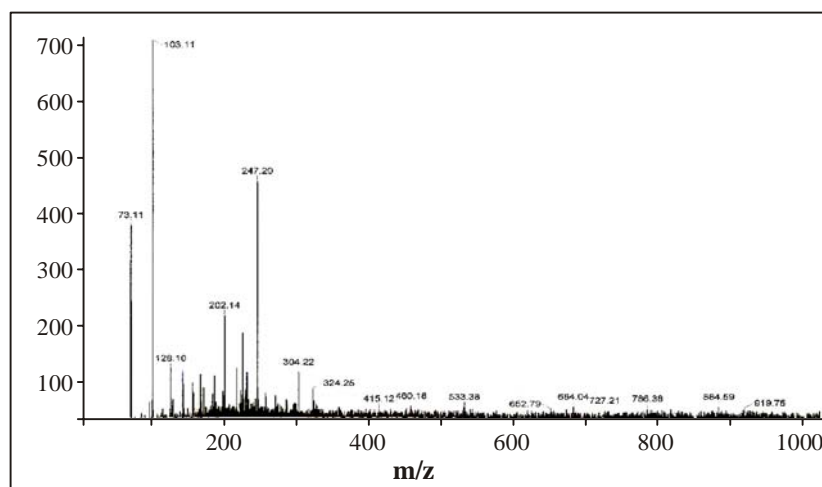


Fig. 1: IR Spectrum of compound

**Table 1: IR spectral data**

Frequency of band (cm <sup>-1</sup> )	Assigned bands	Force constants (Dyne/cm <sup>2</sup> )
3427.0 (b)	=NH	7.87 x 10 <sup>5</sup>
2363.1 (s)	-P=N	6.5 x 10 <sup>5</sup>
1651.9 (b,s)	CO	3.21 x 10 <sup>5</sup>
1405.0 – 1466.2 (w,b)	PO	–
672.1 – 543.0 (b)	-PN-	–

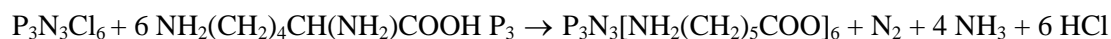
**Fig. 2: Mass spectrum of compound****Table 2: Mass spectral data**

m/z	Fragments	Remarks
73	P <sub>2</sub> N (M-3)	
103	P <sub>2</sub> N <sub>3</sub> (M-3)	Base line peak
128	C <sub>3</sub> H <sub>4</sub> PN <sub>2</sub> O <sub>2</sub> (M-3)	
202	C <sub>6</sub> H <sub>13</sub> PN <sub>2</sub> O <sub>2</sub> (M-3)	
247	C <sub>6</sub> H <sub>13</sub> P <sub>3</sub> N <sub>3</sub> O <sub>2</sub> (M-3)	
304	(CHCOO) <sub>3</sub> N <sub>3</sub> P <sub>3</sub> (M-3)	
533	(CHCOO) <sub>3</sub> (NH <sub>2</sub> CH <sub>2</sub> COO) <sub>3</sub> P <sub>3</sub> N <sub>3</sub> (M-3)	
919	[NH <sub>2</sub> (CH <sub>2</sub> ) <sub>3</sub> PCOO] <sub>6</sub> P <sub>3</sub> N <sub>3</sub> (M-3)	Parent peak

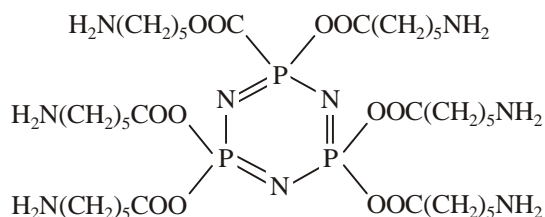
## RESULTS AND DISCUSSION

- (i) On the basis of quantitative estimations, % found, are C-46.32, N-13.7, O-20.98 and H-7.86 and molecular weight is  $915 \text{ mol}^{-1}$ . The compound may be assigned the structure (Fig. 3), which was supported by peak ( $m/z$  -919) in its mass spectrum and microanalysis.
- (ii) This compound formation is supported by frequencies observed in its IR spectrum having the frequencies at  $543.0\text{-}672.1(\text{b})^{22}$ ,  $1405.0\text{-}1466.2$  (w,b),  $1651.9$  (b,s),  $2363.1$  (s) and  $3427.0$  (b) (Table 1), corresponding to five -P-N-, P-O, CO, -P = N and NH bands. The occurrence of vibrations for P-O and C-O linked to P-O group indicates that  $\text{P-Cl}_2$  has reacted with COOH group of amino acid (lysine) through oxygen, due to affinity of phosphorous to oxygen. The value of force constant inferred the P-N and P = N groups
- (iii) Mass spectroscopy of this compound has supported the structure of compound as  $(\text{NP})_3 [\text{OOC}(\text{CH}_2)_5\text{NH}_2]_6$ . The most probable fragments are given in Table 2.

The formation of adduct may be explained by the following reaction -



Hence, the structure of the adduct may be expressed as -



**Fig. 3: Proposed structure of the compound  $\text{P}_3\text{N}_3[\text{NH}_2(\text{CH}_2)_5\text{COO}]_6$**

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