



# **MASS AND I.R. SPECTRAL CHARACTERIZATION OF THE REACTION PRODUCT OF HEXAHYDROXY- CYCLOTRIPHOSHAZENE WITH SALICYLIC ACID**

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## **ABSTRACT**

Salicylic acid was mixed with hexahydroxycyclotriphosphazene,  $[\text{NP}(\text{OH})_2]_3$  in presence of conc.  $\text{H}_2\text{SO}_4$  in alcohol and refluxed for 6 h. The product, obtained, was analyzed, quantitatively, as well as, mass and I. R. spectrometrically and formulated as  $\text{P}_3\text{N}_3[(\text{CO})_3\text{O}_6(\text{C}_6\text{H}_4)_3]$  on the basis of chemical data.

**Key words:** Synthesis, Hexahydroxycyclotriphosphazene.

## **INTRODUCTION**

$(\text{NP}(\text{Cl}_2)_3)$ , and  $(\text{NPH}_2)_3$  have used as ligand and their complexes with metals have been reported<sup>1-9</sup>. The reaction products of  $[\text{NP}(\text{OH})_2]_3$  with acrylic acid, cinnamic acid and oleic acid have also been synthesized and investigated<sup>10</sup>. Therefore, the compounds of  $[\text{NP}(\text{OH})_2]_3$  with salicylic acid was prepared and its studies are being reported here with.

## **EXPERIMENTAL**

Hexahydroxycyclotriphosphazene,  $[\text{NP}(\text{OH})_2]_3$  was synthesized by the reaction of NaOH on  $[\text{NP}(\text{Cl}_2)_3]$  by using Anala R grade chemicals. The product,  $[\text{NP}(\text{OH})_2]_3$  was mixed with salicylic acid (1 : 1 ratio) in alcohol followed by the addition of 1 mL conc.  $\text{H}_2\text{SO}_4$  and refluxed for 6 h, until completion of reaction. The mass formed, was filtered, washed with alcohol and ether successively, dried and stored in a vacuum desiccator over fused  $\text{CaCl}_2$ .

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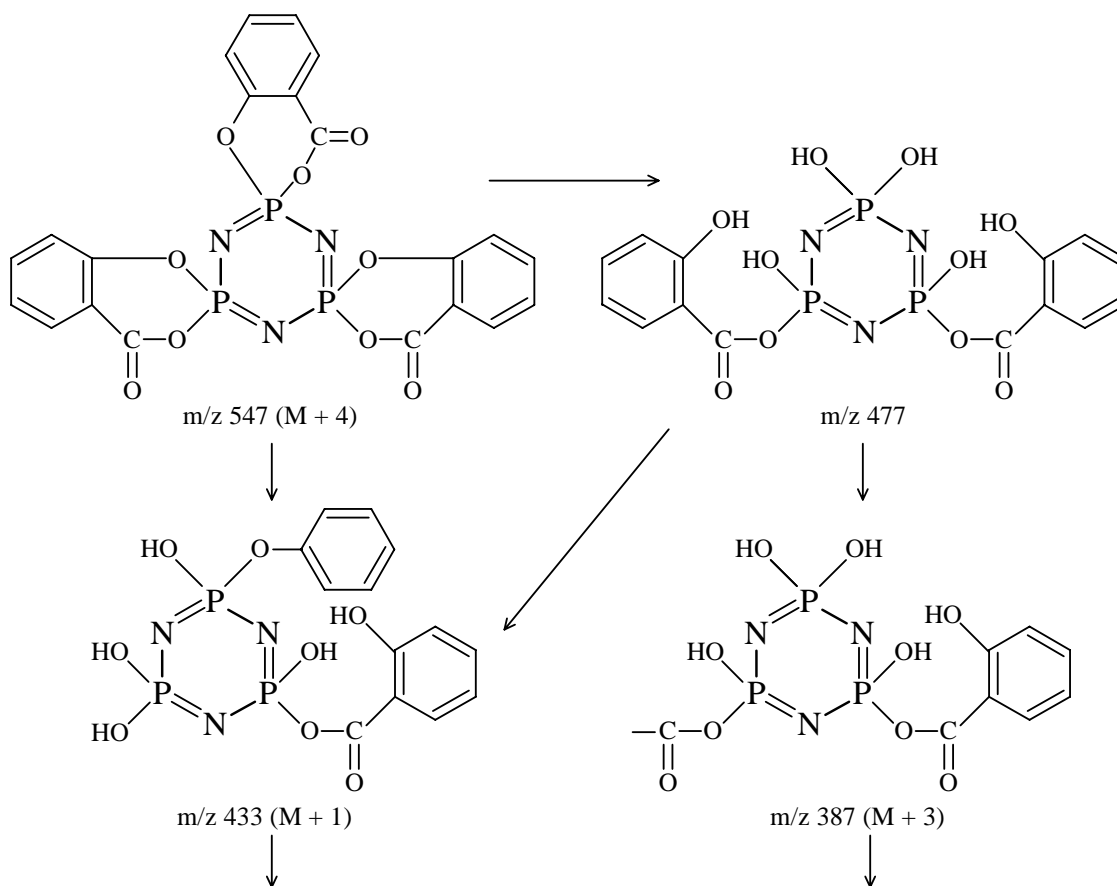
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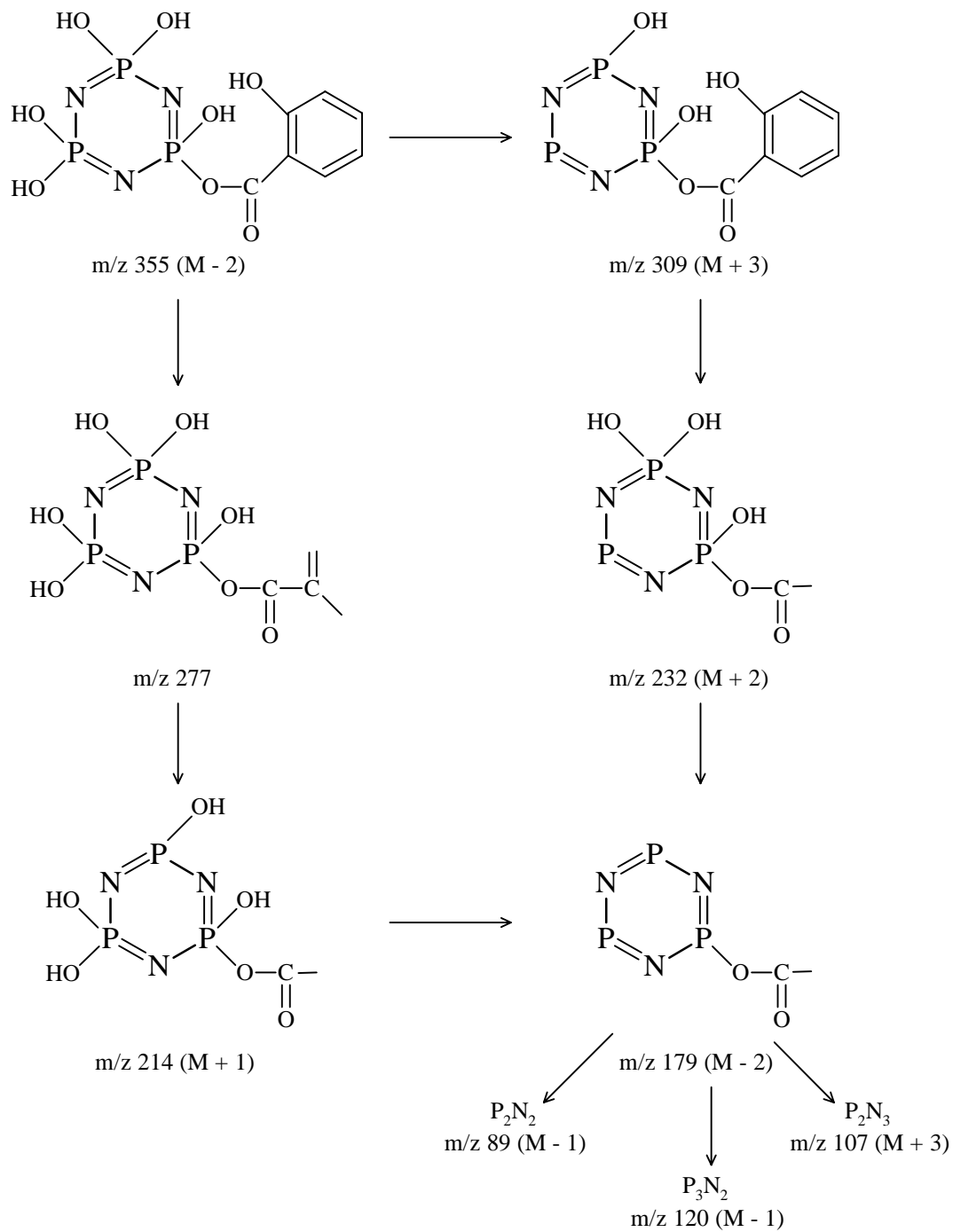
The quantitative estimations for the C, H, N was done at the CDRI, Lucknow. The mol. wt. was determined by Rast's method. Mass and I.R. spectra were carried out on Jeol SX-102 (FAB) and Shimadzu 8201 PC (4000-400  $\text{cm}^{-1}$ ) spectrometers, respectively.

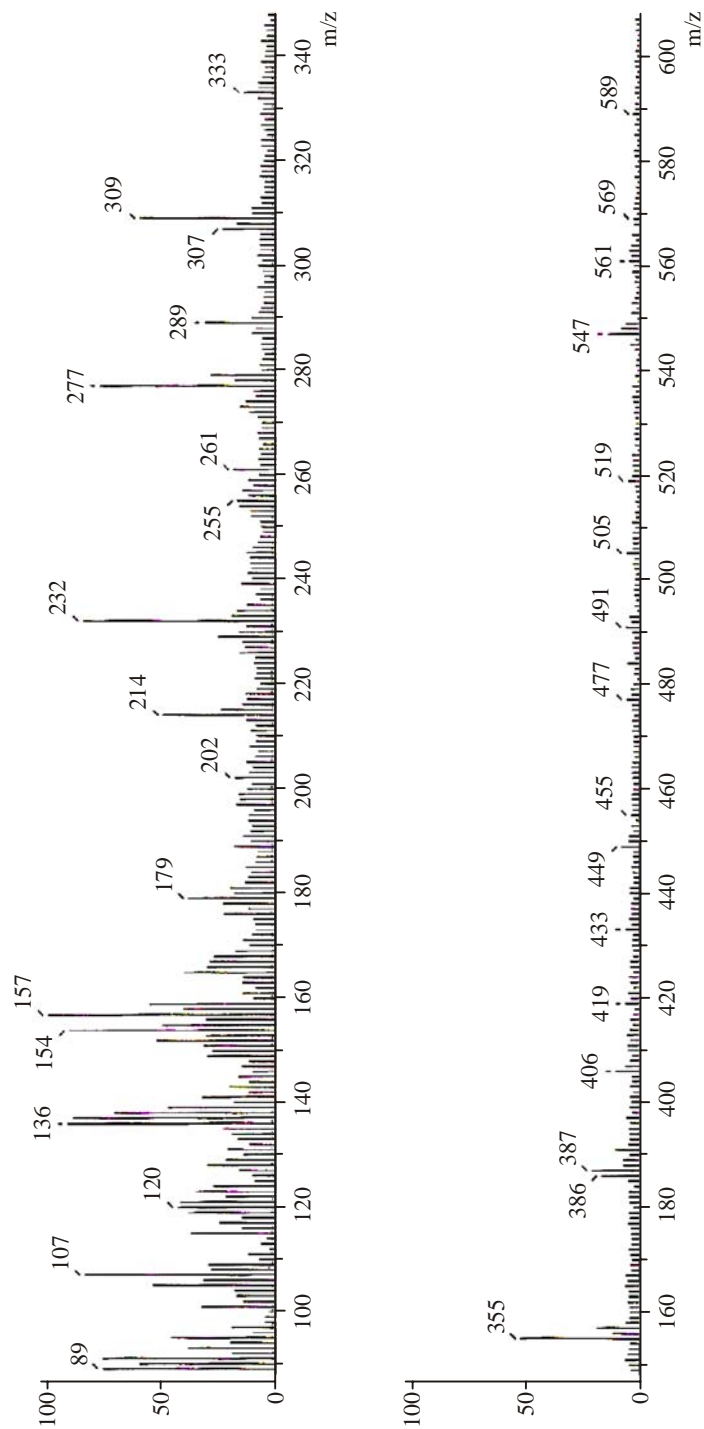
## RESULTS AND DISCUSSION

The compound is blakish brown, soluble in 1,4-dioxan and DMSO. It decomposes in water. The test for N and P as  $\text{NH}_4^+$  and  $\text{PO}_4^{3-}$  ions were found positive.

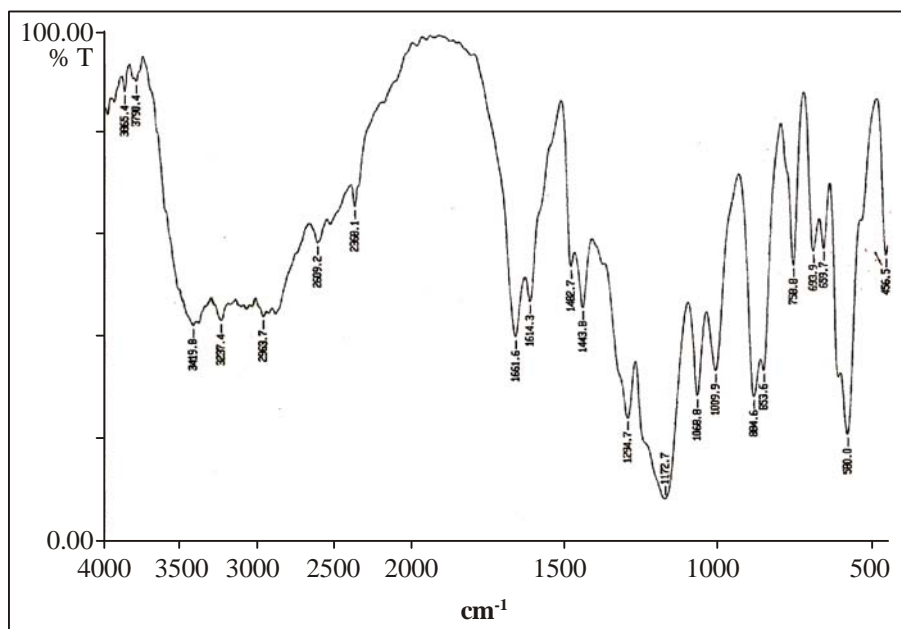
On the basis of the quantitative estimations, % found (Calc.) P 17.04 (17.13), N 7.70 (7.73), C 46.17 (46.41), H 2.20 (2.20), O 26.38 (26.52) and mol. wt. 545.80 (543.0)  $\text{g mol}^{-1}$ , the adduct is formulated as in Fig. 3, which is supported by the mass line  $m/z$  547 ( $M + 4$ ) observed in its mass spectrum (Fig. 1). The other mass lines in the mass spectrum may be illustrated by FAB fragmentation process as follows -



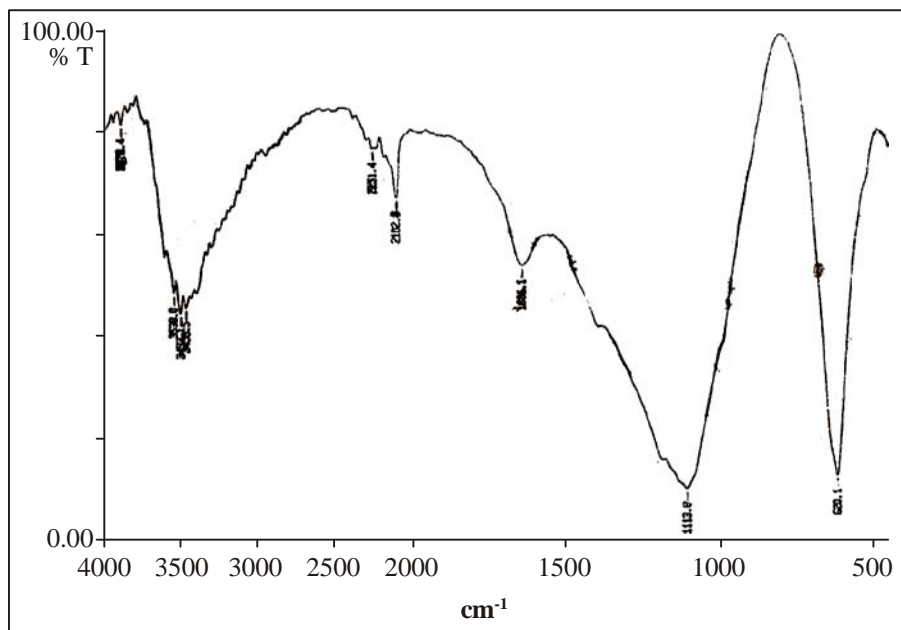


**Fig. 1: Mass spectrum of compound**

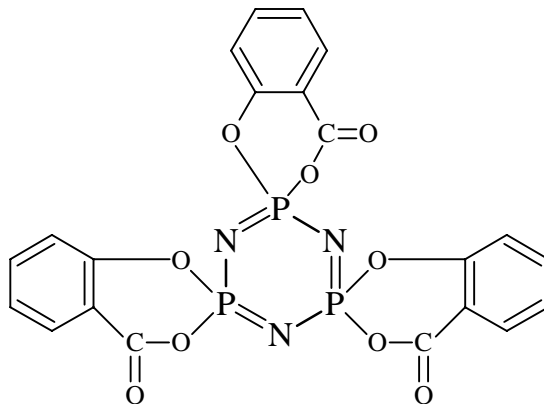
To confirm formation of the compound, its I.R. spectrum (Fig. 2) was compared to



**Fig. 2 (a): IR spectrum of compound**

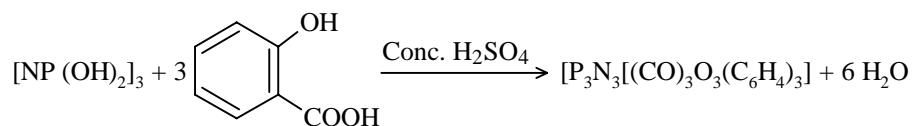


**Fig. 2 (b): IR spectrum of ligand**



**Fig. 3: Structure of complex**

that of  $[\text{NP}(\text{OH})_2]_3$ , ligand. The vibrations, observed at  $456.5\text{--}853.6\text{ cm}^{-1}$  and  $884.6\text{ cm}^{-1}$  are for P-N band and P-O band, respectively as in  $[\text{NP}(\text{OH})_2]_3$ . Two vibrations at  $1009.9\text{ cm}^{-1}$  and  $1068.8\text{ cm}^{-1}$  for  $\text{C}_6\text{H}_5\text{--OH}$  band indicates its presence in the compound. The peaks were observed at  $1172.7\text{ to }1294.7\text{ cm}^{-1}$  and  $1443.8\text{ to }1482.7\text{ cm}^{-1}$  for  $\text{O}=\text{P}\text{--OH}$  group and  $\text{C}=\text{C}\text{--H}$  band, respectively. The frequencies at  $1614.3\text{ to }2368.1\text{ cm}^{-1}$  for  $\text{C}=\text{O}$  group (Ketonic group) linked to  $\text{C}=\text{C}$  and for  $\text{--COOH}$  group at  $2609.2\text{ to }2963.7\text{ cm}^{-1}$  also appeared in I.R. spectrum of the compound, which are due to salicylic acid. The band at  $3237.4\text{ to }3419.8\text{ cm}^{-1}$  as in  $[\text{NP}(\text{OH})_2]_3$  for P-OH linkage have been found. Thus from the results, it is confirmed that salicylic acid has reacted with  $[\text{NP}(\text{OH})_2]_3$  in presence of conc.  $\text{H}_2\text{SO}_4$  with the elimination of  $\text{H}_2\text{O}$  molecules forming the compound as follow:



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