



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

ATUL GUPTA and S. P. S. JADON*

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

ABSTRACT

Salicylic acid was mixed with hexahydroxycyclotriphosphazene, $[\text{NP}(\text{OH})_2]_3$ in presence of conc. H_2SO_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 reported¹⁻⁹. The reaction products of $[\text{NP}(\text{OH})_2]_3$ with acrylic acid, cinnamic acid and oleic acid have also been synthesized and investigated¹⁰. 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. H_2SO_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 CaCl_2 .

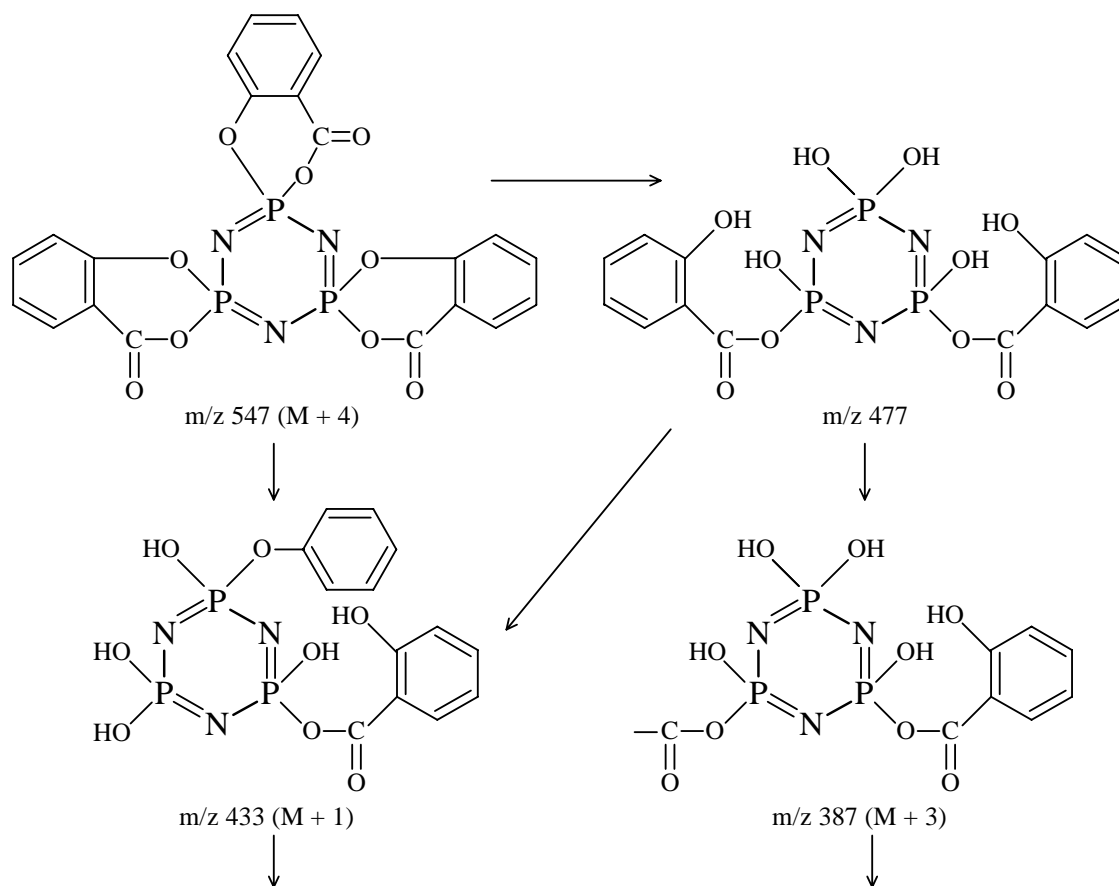
* Author for correspondence; E-mail: sps_jadon@yahoo.co.in

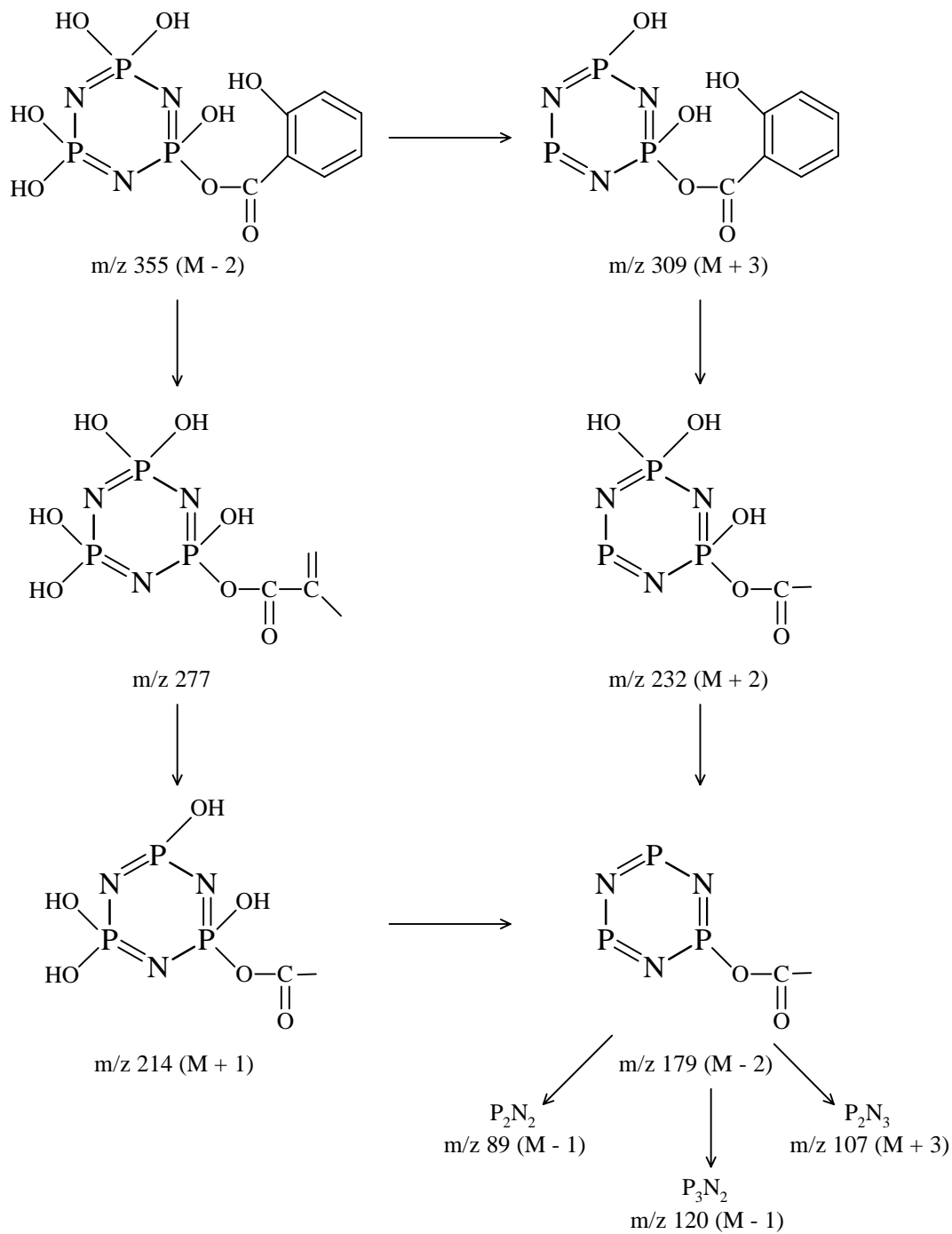
The quantitative estimations for the C, H, N was done at the CDRI, Lucknow. The mol. wt. was determined by Rast's method. Mass and I.R. spectra were carried out on Jeol SX-102 (FAB) and Shimadzu 8201 PC (4000-400 cm^{-1}) spectrometers, respectively.

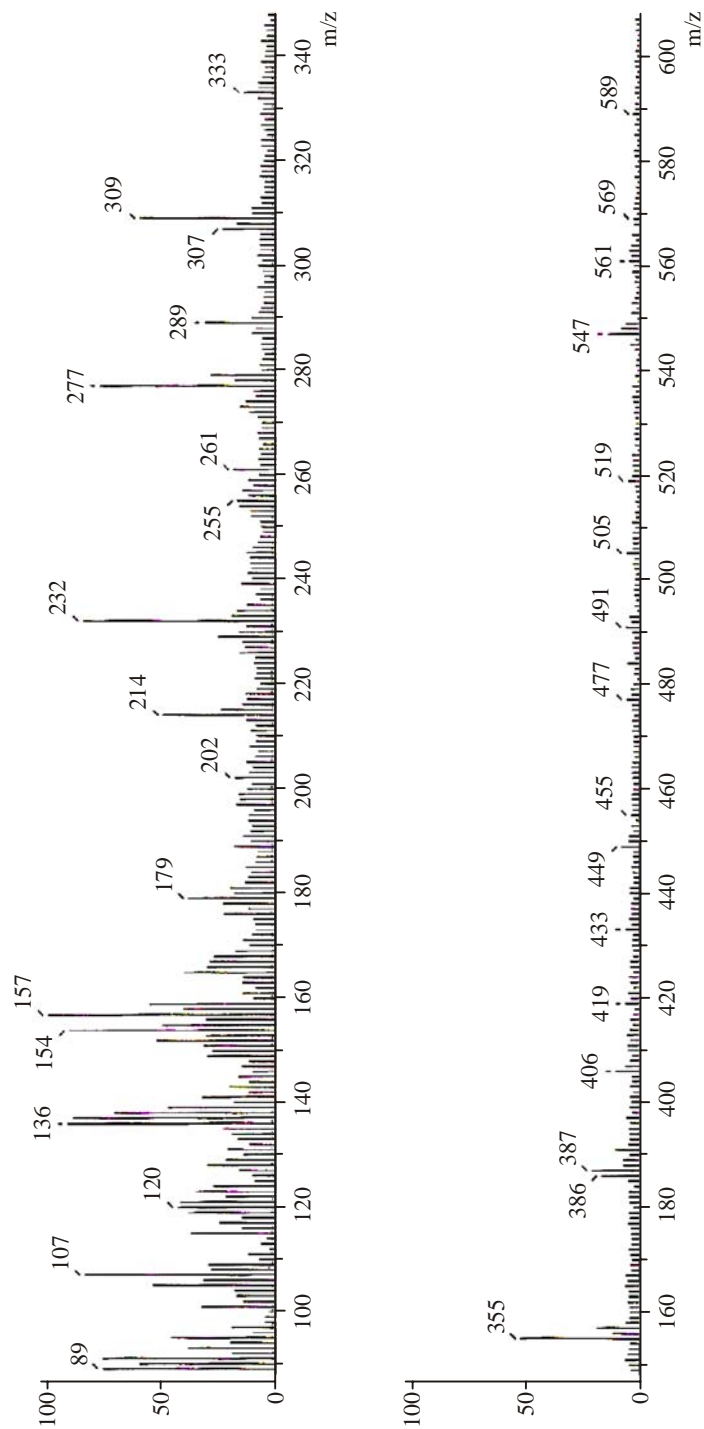
RESULTS AND DISCUSSION

The compound is blakish brown, soluble in 1,4-dioxan and DMSO. It decomposes in water. The test for N and P as NH_4^+ and 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) 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

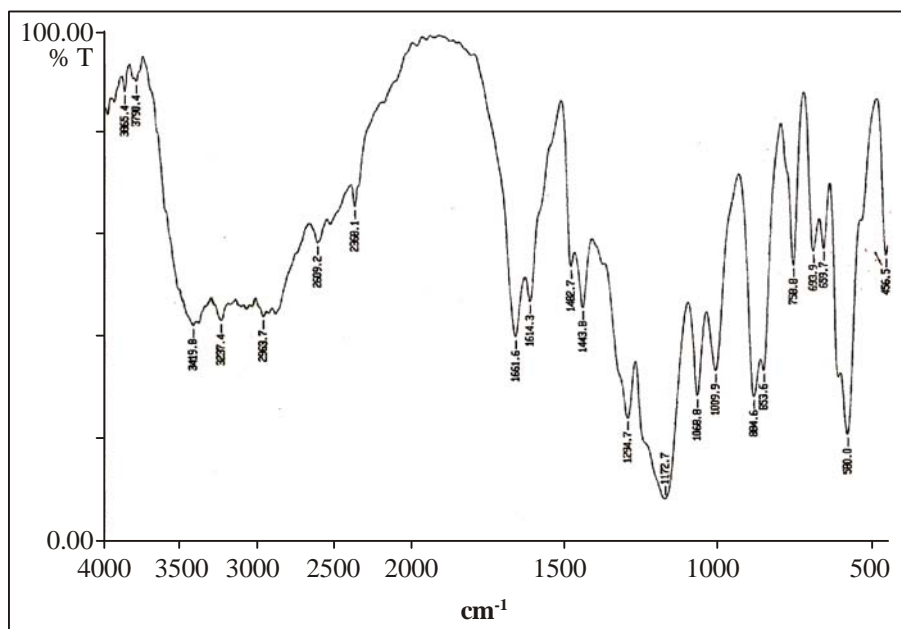


Fig. 2 (a): IR spectrum of compound

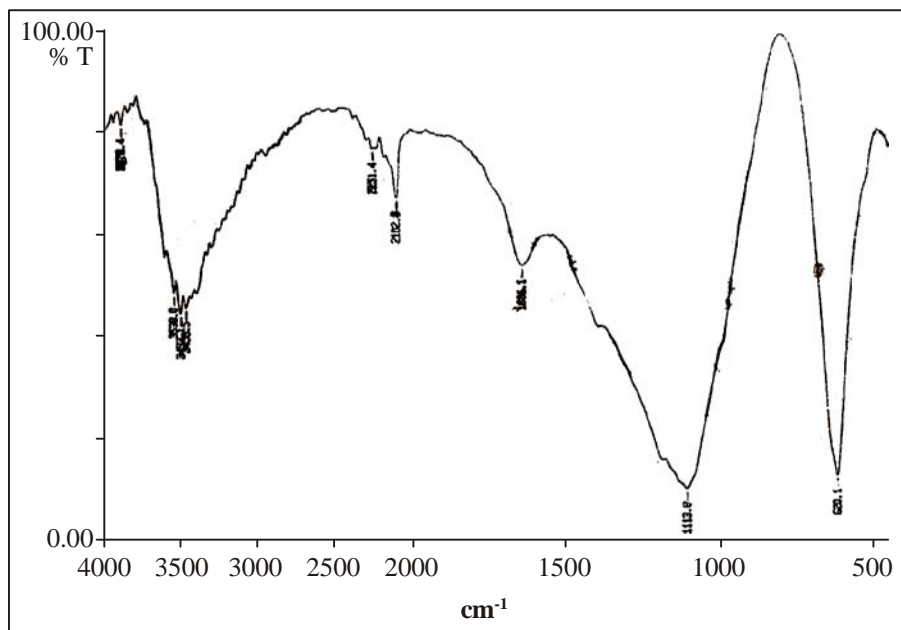


Fig. 2 (b): IR spectrum of ligand

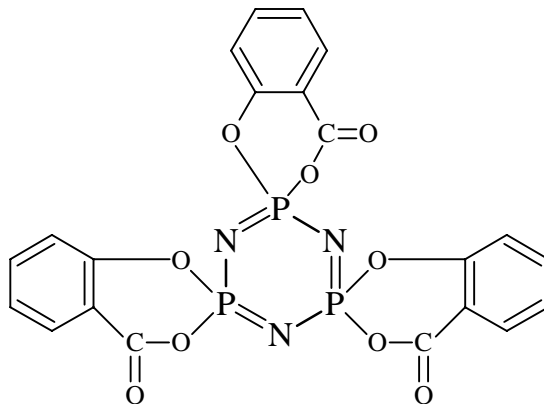
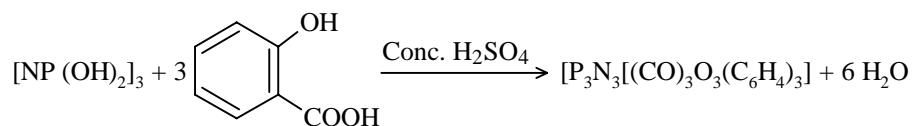


Fig. 3: Structure of complex

that of $[\text{NP}(\text{OH})_2]_3$, ligand. The vibrations, observed at $456.5\text{--}853.6\text{ cm}^{-1}$ and 884.6 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 cm^{-1} and 1068.8 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 --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. H_2SO_4 with the elimination of H_2O molecules forming the compound as follow:



ACKNOWLEDGEMENT

Authors wishes to thank to the Director, C.D.R.I., Lucknow for providing instrumental facilities.

REFERENCES

1. H. Binder, Z. Inorg. Alleg. Chem. (Gen.) **383**, 130 (1971).
2. Y. Busleav, B. V. Levin, M. Z. G. Ry, S. P. Petrosynnts and B. V. Micronova, Zh. Neorg. Khim., **14**, 3245 (1969).

3. H. W. Raesky and H. Weizer, *Ber.*, **106**, 280 (1973).
4. H. R. Slock, *Inorg. Chem.*, **38**, 280 (1999).
5. O. S. Jung, *Inorg. Chem.*, **38**, 5447 (1999).
6. S. P. S. Jadon, *Asian J. Chem.*, **15**, 151 (2003); **17**, 1312 (2005).
7. A. Sundermann and W. W. Scholler, *Inorg. Chem.*, **38**, 6261 (1999).
8. N. Jain and S. P. S. Jadon, *Asian J. Chem.*, **18**, 730 (2006).
9. N. Jain and S. P. S. Jadon, *Int. J. Chem. Sci.*, **4**, 285 (2006).
10. Illa Rani and S. P. S. Jadon, *Asian J. Chem.*, **20(7)**, 5711 (2008); *Int. J. Chem. Sci.*, **6(2)**, 519 (2008).

Accepted : 21.08.2011