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

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ABSTRACT

Salicylic acid was mixed with hexahydroxycyclotriphosphazene, $[NP(OH)_2]_3$ 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 $P_3N_3[(CO)_3O_6(C_6H_4)_3]$ on the basis of chemical data.

Key words: Synthesis, Hexahydroxycyclotriphosphazene.

INTRODUCTION

$(NPCl_2)_3$, and $(NPH_2)_3$ have used as ligand and their complexes with metals have been reported[1-9]. The reaction products of $[NP(OH)_2]_3$ with acrylic acid, cinnamic acid and oleic acid have also been synthesized and investigated[10]. Therefore, the compounds of $[NP(OH)_2]_3$ with salicylic acid was prepared and its studies are being reported here with.

EXPERIMENTAL

Hexahydroxycyclotriphosphazene, $[NP(OH)_2]_3$ was synthesized by the reaction of NaOH on $[NP(Cl_2)]_3$ by using Anala R grade chemicals. The product, $[NP(OH)_2]_3$ 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 $CaCl_2$.

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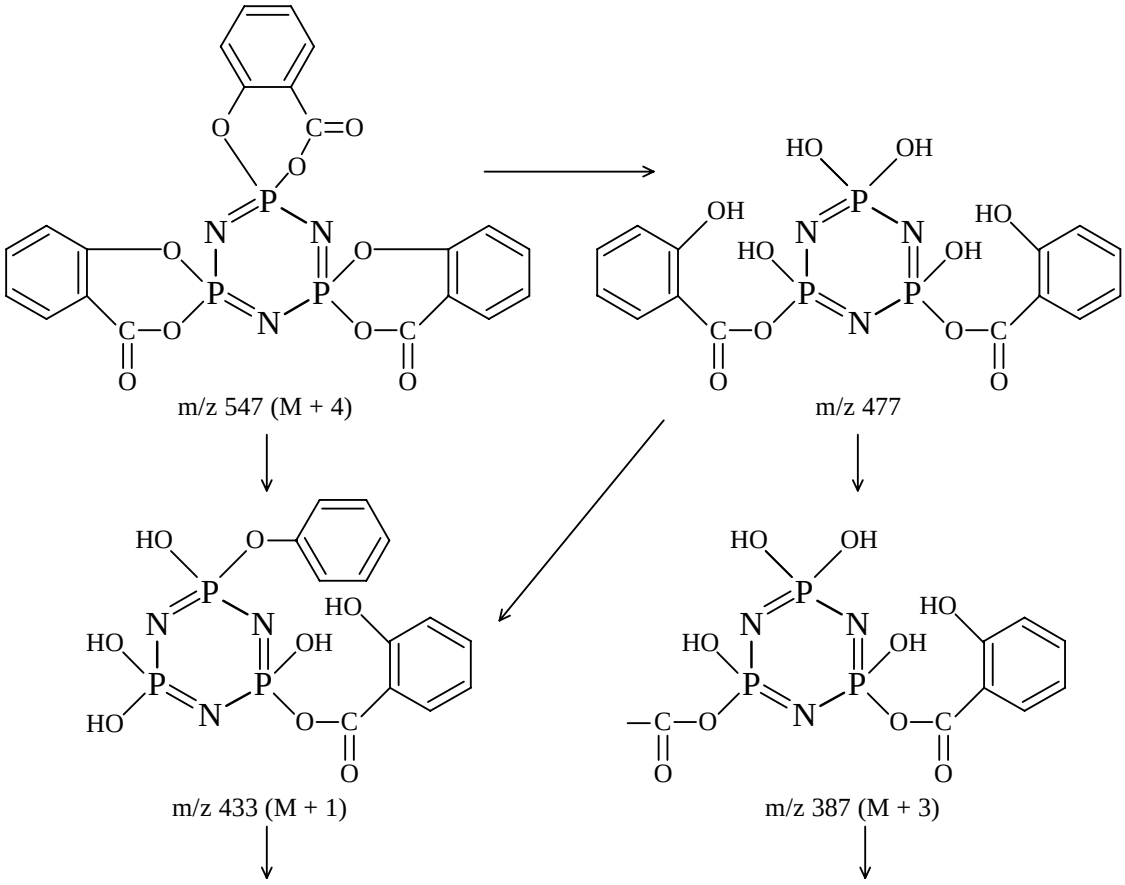
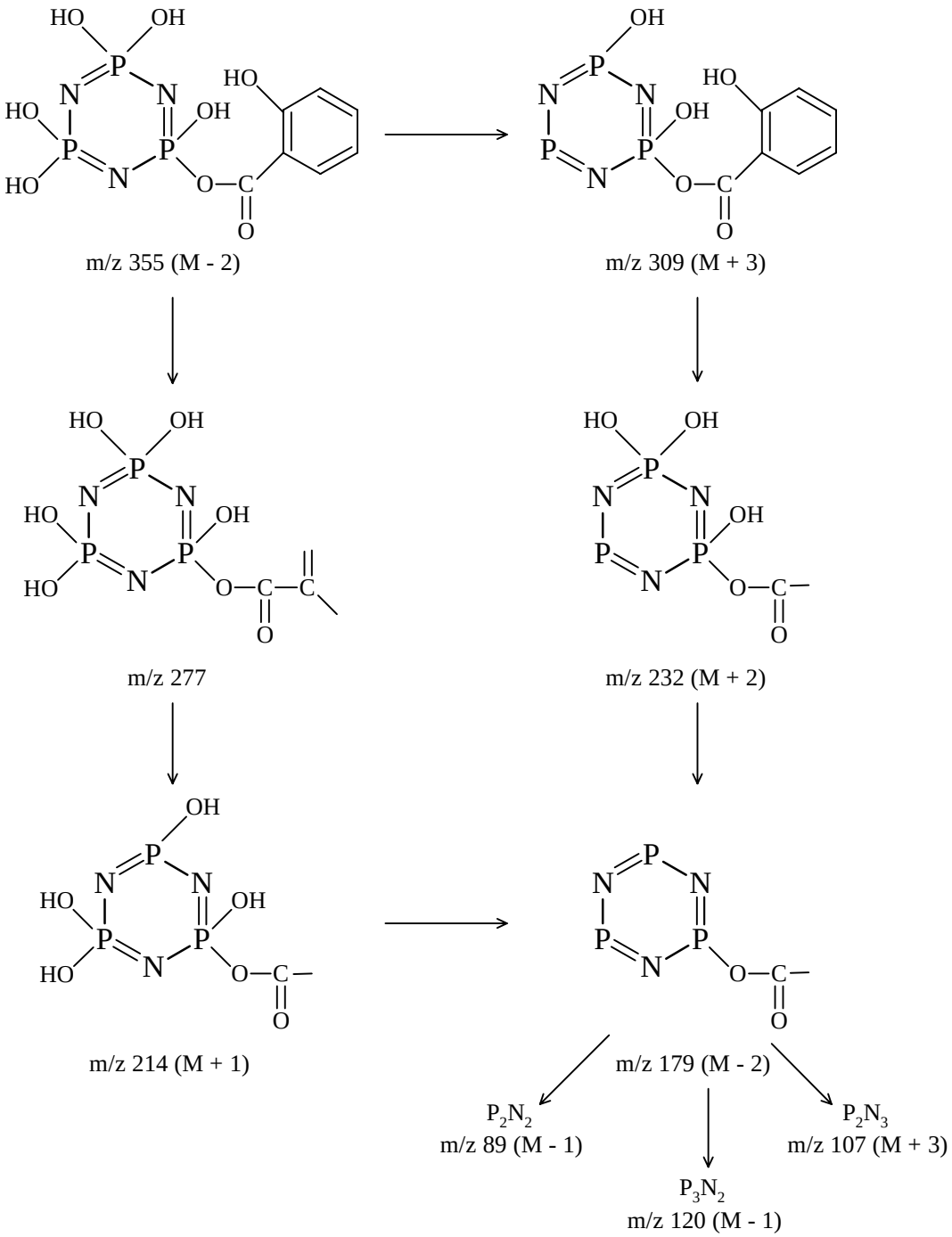
The quantitative estimations for the C, H, N was done at the CDRI, Lucknow. The mol. wt. was determined by Rast's method. Mass and I.R. spectra were carried out on Jeol SX−102 (FAB) and Shimadzu 8201 PC (4000-400 cm^{-1}) spectrometers, respectively.

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 -




m/z 355 (M - 2)
m/z 309 (M + 3)
m/z 277
m/z 232 (M + 2)
m/z 214 (M + 1)
m/z 179 (M - 2)
P_2N_2
m/z 89 (M - 1)
P_2N_3
m/z 107 (M + 3)
P_3N_2
m/z 120 (M - 1)

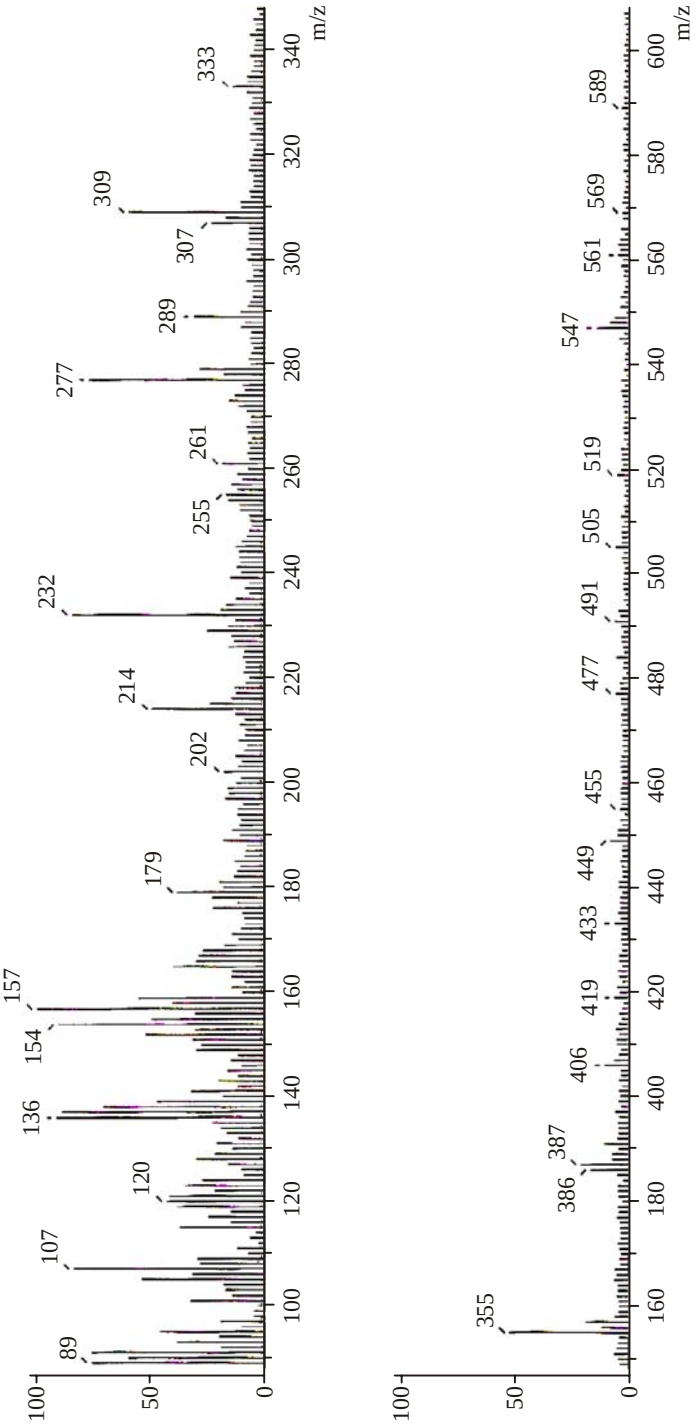


Fig. 1: Mass stpectrum of compound

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

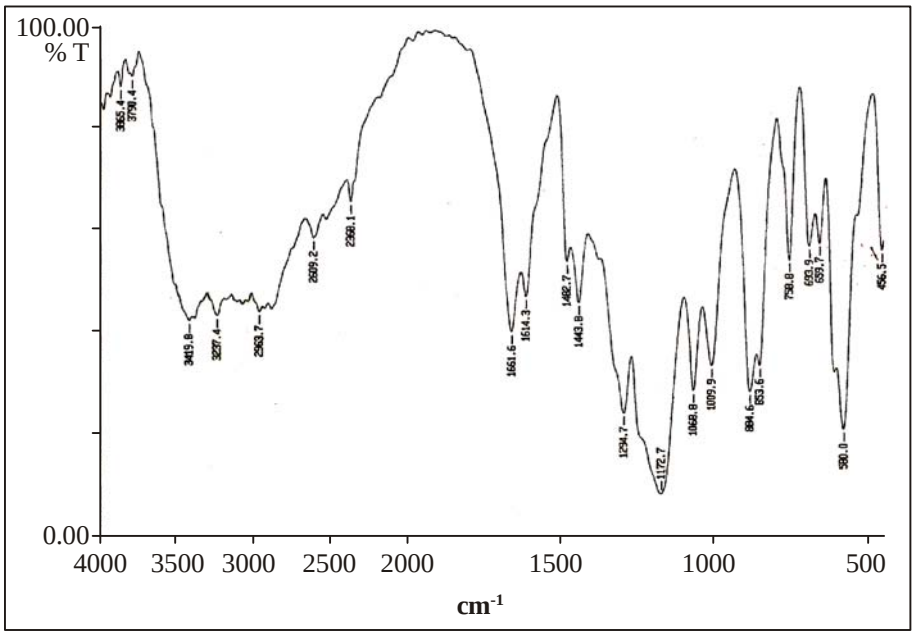


Fig. 2 (a): IR spectrum of compound

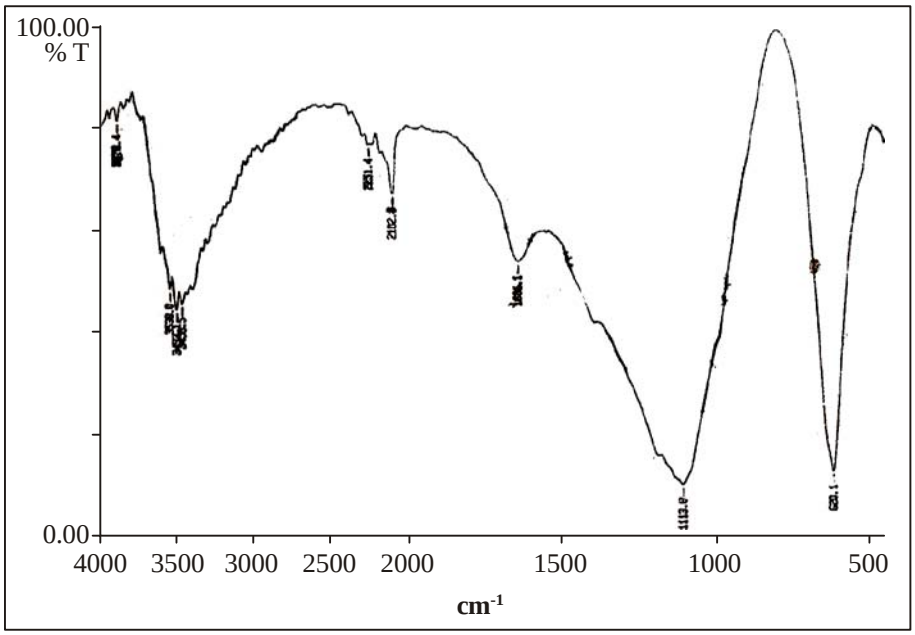


Fig. 2 (b): IR spectrum of ligand

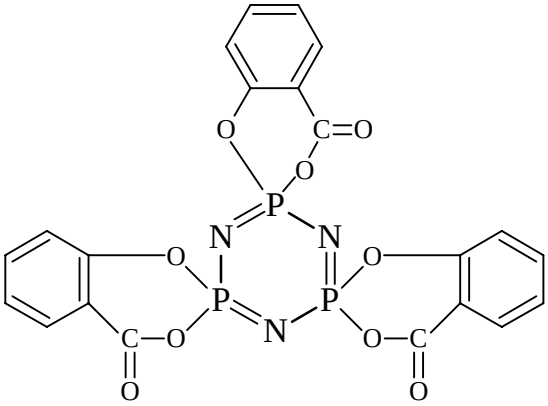
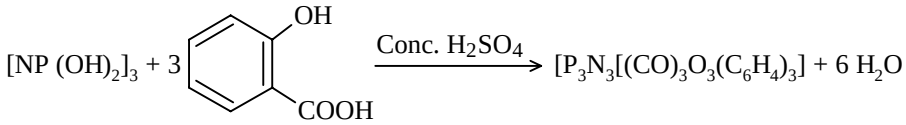


Fig. 3: Structure of complex

that of $[NP(OH)_2]_3$, ligand. The vibrations, observed at 456.5-853.6 cm^{-1} and 884.6 cm^{-1} are for P-N band and P-O band, respectively as in $[NP(OH)_2]_3$. Two vibrations at 1009.9 cm^{-1} and 1068.8 cm^{-1} for C_6H_5–OH band indicates its presence in the compound. The peaks were observed at 1172.7 to 1294.7 cm^{-1} and 1443.8 to 1482.7 cm^{-1} for O = P –OH group and C = C – H band, respectively. The frequencies at 1614.3 to 2368.1 cm^{-1} for C = O group (Ketonic group) linked to C = C and for –COOH group at 2609.2 to 2963.7 cm^{-1} also appeared in I.R. spectrum of the compound, which are due to salicylic acid. The band at 3237.4 to 3419.8 cm^{-1} as in $[NP(OH)_2]_3$ for P-OH linkage have been found. Thus from the results, it is confirmed that salicylic acid has reacted with $[NP(OH)_2]_3$ in presence of conc. H_2SO_4 with the elimination of H_2O molecules forming the compound as follow:



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