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Removal of Nicotinic Acid by $[NP(OH)_2]_3$ and Identification of product by Its Mass, IR and NMR Spectra

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ABSTRACT

The adduct of $[NP(OH)_2]$ with nicotinic acid synthesized was analysed qualitatively, quantitatively, mass, I.R. and ^{1}HNMR spectrometrically. The cream coloured adduct having the molecular formula, $(P_3N_3)(C_5H_3N)_4(OH)_{10}(CO)_4O_4]$ is soluble in water suggesting as Nicotinic acid present in the body may be removed by the hexahydroxyphosphazene.

Keywords: Nicotinic acid, spectra

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INTRODUCTION

$(NPCl_2)_3$ and $(NPH_2)_3$ trimers 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 compound of $[NP(OH)_2]_3$ with nicotinic acid was prepared and its studies are being reported here with.

MATERIALS AND METHODS

Experimental

Hexahydroxycyclotriphosphazene, $[NP(OH)_2]_3$ was synthesized by the reaction of NaOH on $[NP(Cl)_2]_3$ by using Anala R grade chemical [11]. The product, $[NP(OH)_2]_3$ was mixed with nicotinic acid (1:1 ratio) in alcohol following by the addition of 1ml conc. H_2SO_4 and refluxed for 6h until the completion of reaction. The mass formed was filtered, washed with alcohol and ether successively, dried and stored in a vacuum desiccators.

The quantitative estimations for the C, H, and N get done from the CDRI Lucknow.

The mol.wt. was determined by using Rost's process using the equation,

$$(M)=\frac{Kf\times 1000\times w_1}{\Delta T\times w_2}.$$

Mass, I.R. and ^{1}HNMR spectra were carried out subsequently on Jeol SX-102 (FAB), Shimadzu 8201 PC (4000-400cm^{-1}) and Bruker DRX-300 spectrometers at room temperature.

RESULTS AND DISCUSSION

The cream coloured adduct of $[NP(OH)_2]_3$ with nicotinic acid is soluble in water. The presence of N & P was confirmed by testing for NH^+_4 and Po_4^{3-} ions.

Molecular formula, $[(P_3N_3)_3(C_5H_3N)_4.(OH)_{10}(CO)_4.O_4]$ for adduct was established on the basis of analytical data, % found (cal) P26.29(26.34), N 17.15(17.19), C 27.14(27.19) H 2.07(2.08) O 27.17 (27.19) and mol. wt. 1061 (1059) gm mol^{-1}.

This molecular formula is supported by the mass line m/z 1062 (M+3) which is too much close to the 1061 gm mol^{-1} found by classical method, observed in its mass spectrum (Fig. 1), exploring that three molecules of $P_3N_3(OH)_6$ or $[NP(OH)_2]_3$ has reacted with four molecule of nicotinic acid in presence of conc. H_2SO_4 forming adduct and eliminating water molecules. The reaction may be expressed as follow:

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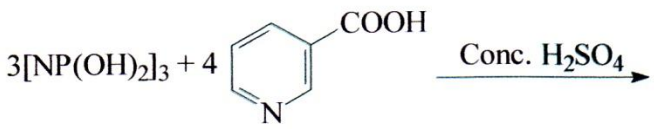
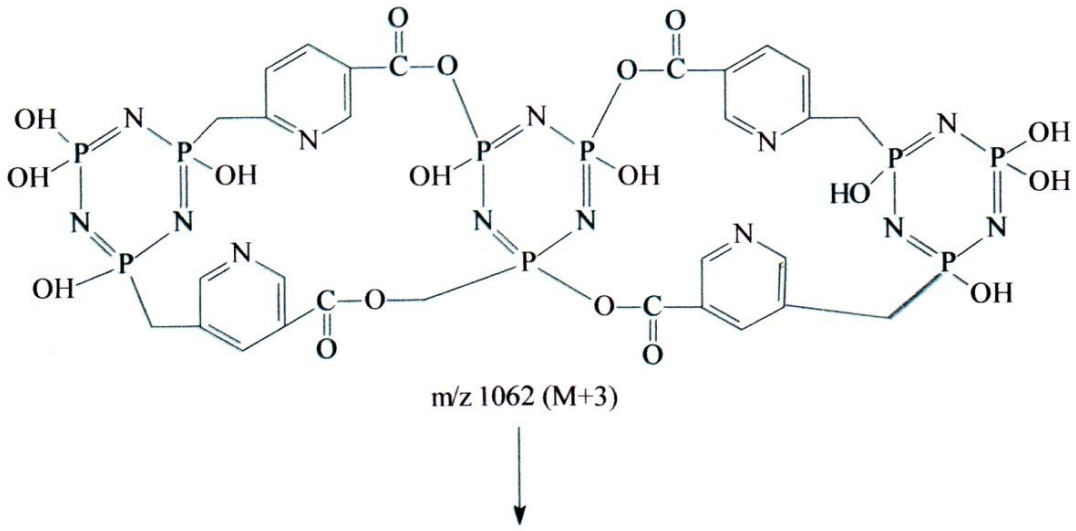


$$(P_3N_3)_3(C_5H_3N)_4(OH)_{10}(CO)_4.O_4 + 8H_2O$$

The other mass lines in its mass spectrum (Fig. 1) may be explained be the fragmentation process.



m/z 1062 (M+3)

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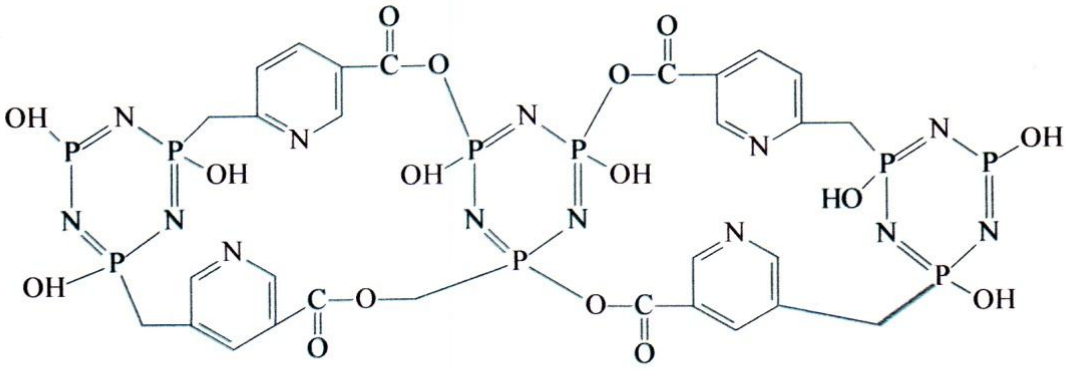
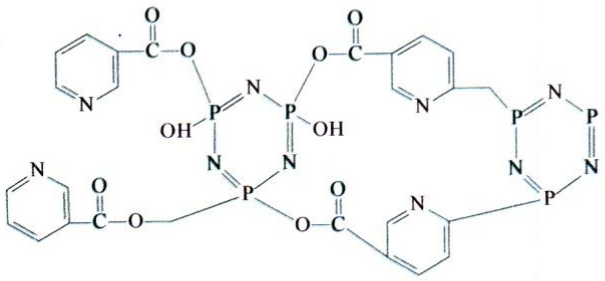
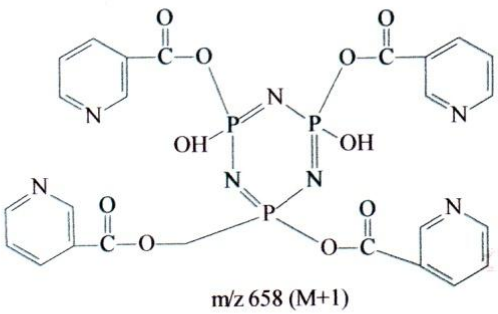


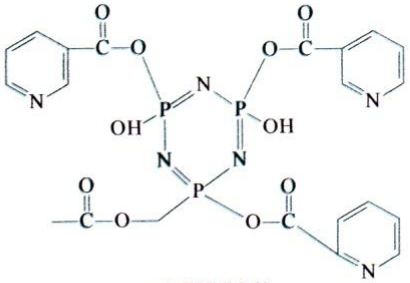
m/z 1025



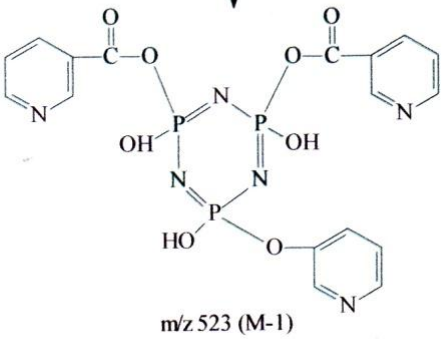
m/z 718 (M-2)



m/z 658 (M+1)



m/z 578 (M-1)



m/z 523 (M-1)

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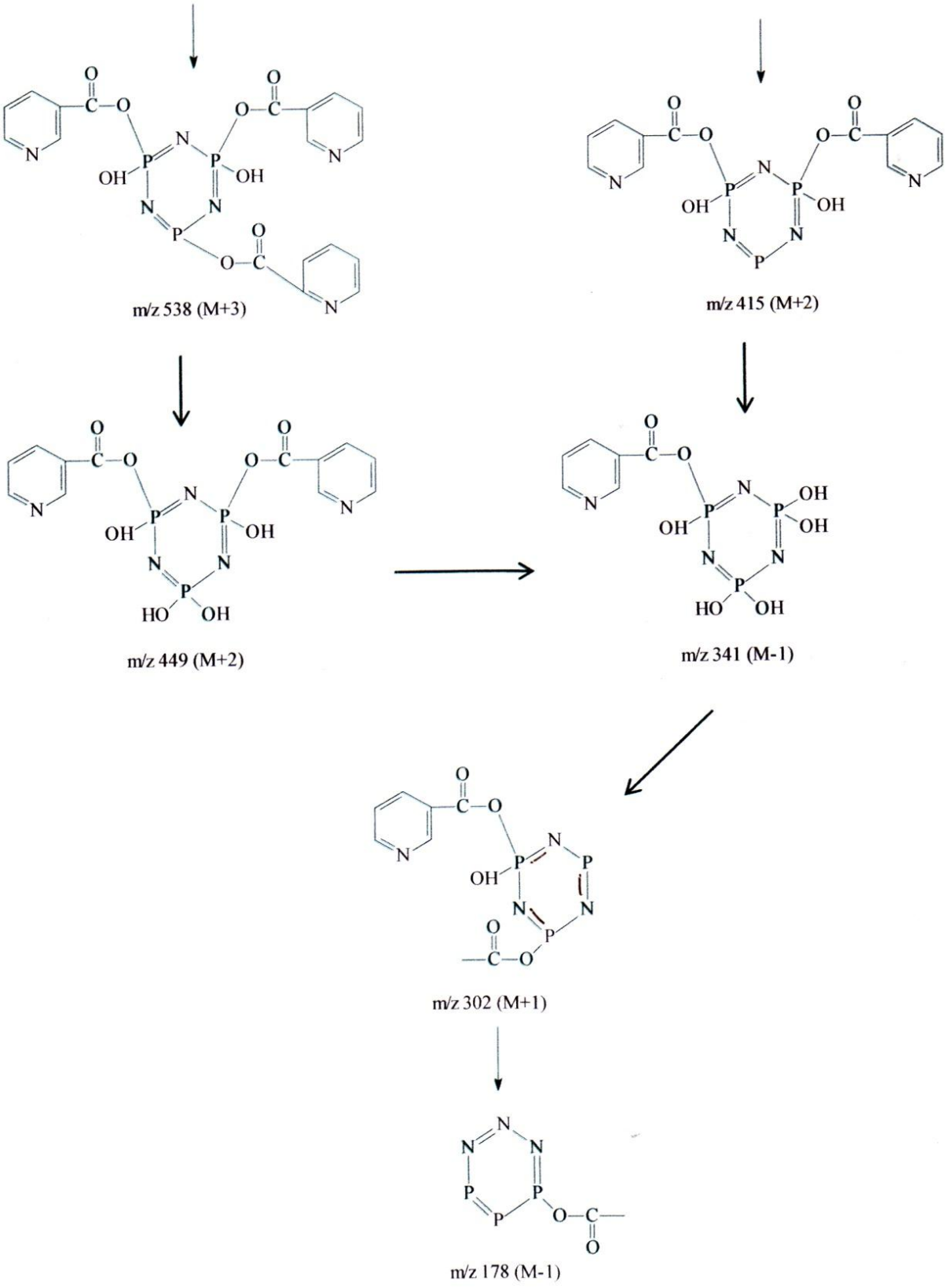

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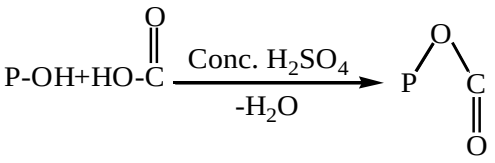
The I.R. spectrum of the adduct is compared is that of ligand Fig.2. The vibration observed in the I.R. spectrum of ligand at 620.1cm^{-1} is slightly lowered to 617.3 cm^{-1} is for P-N bands. The frequency at 1113.8cm^{-1} for P-O gp has also shifted to higher region 1134.2cm^{-1} as a broad peak in the I.R. spectrum of adduct showing the linkage of P-O band to other group. Similarly the band at 1638.1cm^{-1} found in the I.R. spectrum of the ligand is for P-O gp. which has disappeared in the I.R. spectrum of adduct with a new frequency at 1618.6cm^{-1} (w,b) for C=0 gp which has linkage with the P-O band. The vibrations at 2102.8cm^{-1} is also absent in the I.R. spectrum of adduct while the band at 2099.4cm^{-1} (w and doublet) is for the C_5H_3NCO gp. The new vibrations in the I.R. spectrum of the adduct at 2339.6cm^{-1} (broad and w, doublet) 2969.5cm^{-1} for C=N and –COO– bands respectively confirms the presence of nicotinic acid in the adduct. Vibrations at higher region 3458.5cm^{-1} (b) for P-OH gp in the I.R. spectrum of ligand has lowered to 3420.1cm^{-1} showing the reactivity of P-OH gp with the nicotinic acid in the presence of conc. H_2SO_4 expanding the following reaction.



.....(4)

Thus it is confirmed that nicotinic acid has reacted with $(NP(OH)_2)_3$ with the elimination of water molecule in the presence of conc. H_2SO_4 confirming its molecular formula and structure fig. 4.

^{1}HNMR FOR NICOTINIC ACID

^{1}HNMR spectrum (fig. 3) consist 5 sets of signals out of which the set of signals in the range of chemical shift δ1.91 – δ2.124 and δ7.789 – δ8.578 ppm possesses similar number of signals with a mirror image for the P-N ring having same number of four OH groups in opposite direction. The set of signals in the range of δ 3.556 – δ 4.003ppm are for a P-N ring having two OH groups linked to opposite P atoms of the ring. The remaining sets of signals in the range of δ2.216 – δ3.340 and δ6.552 to δ7.789ppm are for the four nicotinic acid groups having face to face N atoms and linkage to two P-N ring in opposite direction as explored by the its structure Fig. 4.

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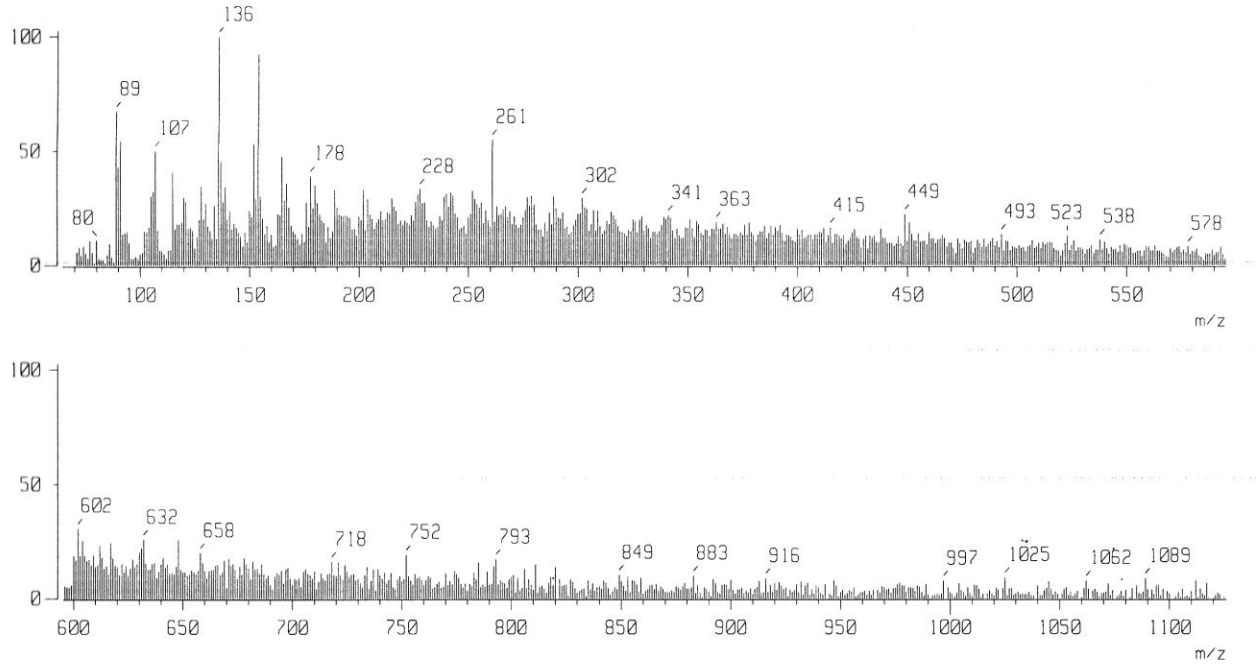
Fig. 1 – Mass Spectrum of Compound



→ Wave Length (cm^{-1})-

Fig. 2: IR spectrum (a) Ligand (b) Compound

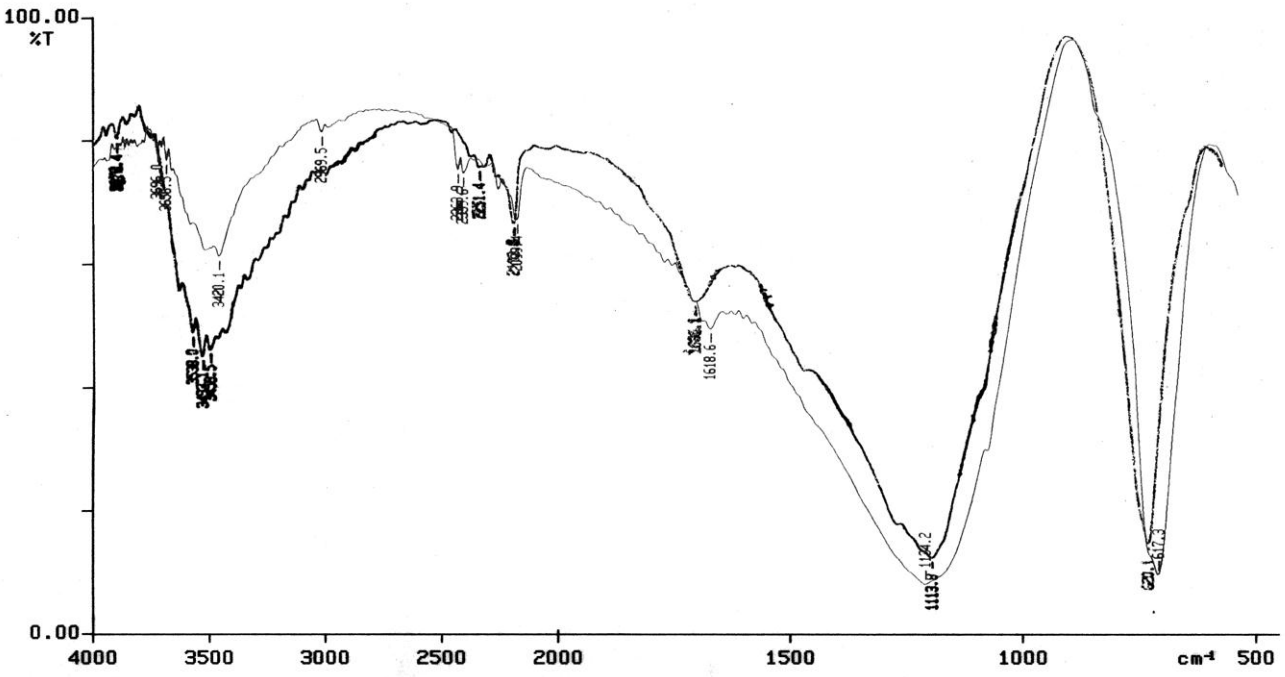


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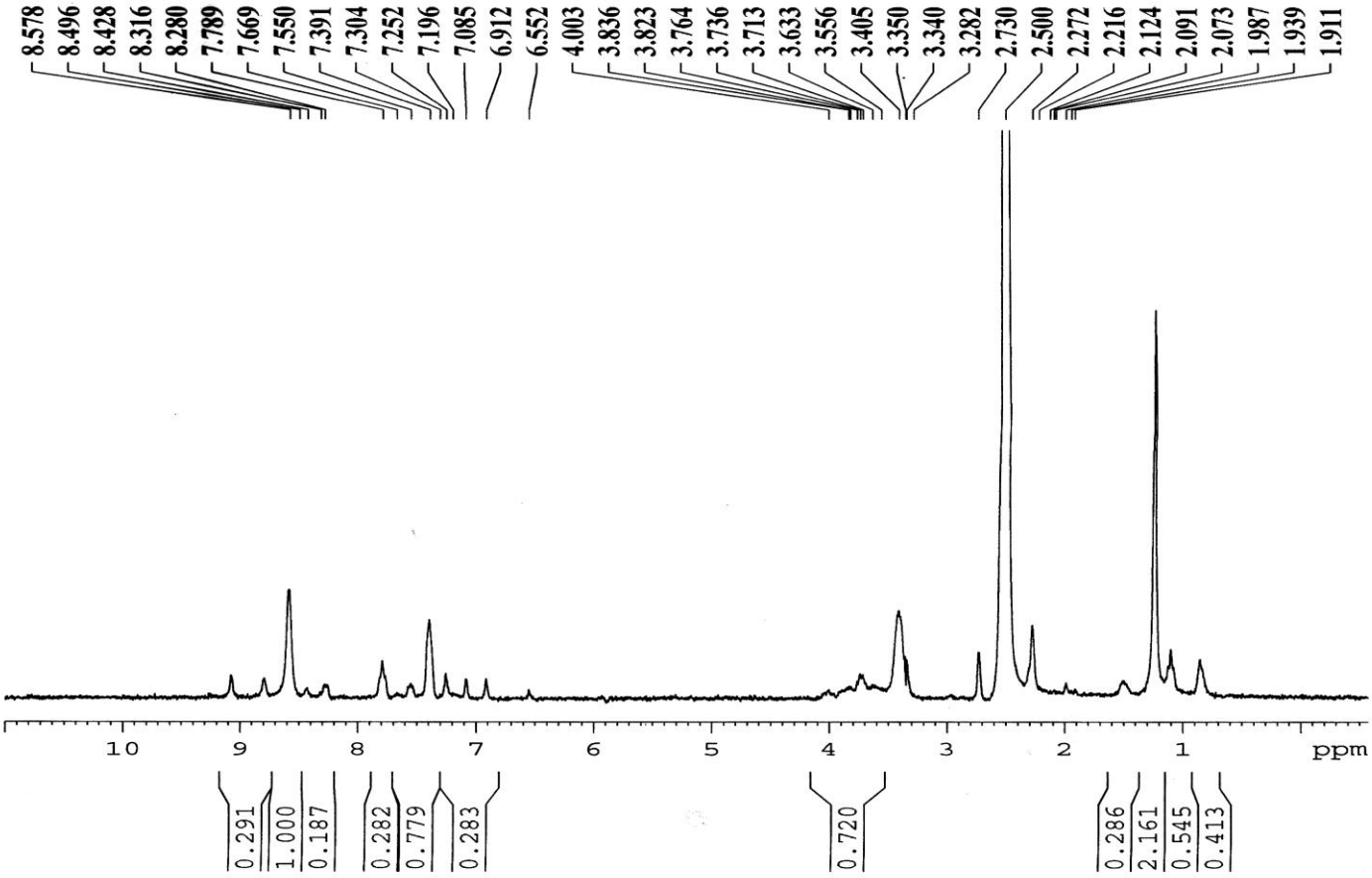
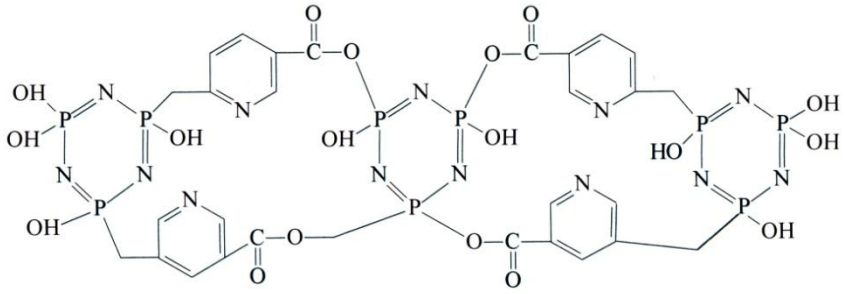
Fig. 3: ^{1}H NMR spectrum of Compound





m/z 1062 (M+3)

Fig. 4 – Structure of Adduct, hydroxyphosphazene niconate

CONCLUSION

From the result it is concluded the nicotinic acid reacts with $[NP(OH)_2]_3$ producing $[(P_3N_3)(C_5H_3N)_4(OH)_{10}(CO)_4.O_4]$ which is water soluble compound. From this study it is inferred that nicotinic acid formed in human body may be removed by non-toxic $[NP(OH)_2]_3$ hydroxyphosphazened.

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