

SPECTRAL CHARACTERIZATION OF REACTION PRODUCT OF OLEIC ACID AND HEXAHYDROXYCYCLOPHOSPHOZENE

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ABSTRACT

 $[NP(OH)_2]_3$ was mixed with oleic and refluxed in DMF in presence of conc. H_2SO_4 . The mass, produced, was estimated quantitatively, along with its mass and I. R. spectra. The product has been assigned the structure $[P_3N_3 \ (OH)_4]_4$ - $[O-CO-(CH_2)_7$ - $CH=CH-(CH_2)_8$ - $]_5H_2$ as an open chain polymeric compound.

Key words: Tetrahydroxy tetraphosphozene pentaoleiate, Polymeric, Fragmentation, Condensation.

INTRODUCTION

A few complexes of hexahydroxycyclophosphozene (NPOH₂)₃ have been reported¹⁻⁵, but neither the complexes of [NP(OH)₂]₃ with metals nor its organosub-situted derivatives have been synthesized. Therefore, the condensation reaction between oleic acid and hexahydroxycyclophosphozene, [NP(OH)₂]₃ has been carried out and spectral data of the product obtained have been reported in the present work.

EXPERIMENTAL

The starting material, $(NPCl_2)_3$ was synthesised⁶ and separated from other higher polymers by column chromatography. $[NP(OH)_2]_3$ was prepared by the reaction of NaOH (dry pellet) on $(NPCl_2)_3$ dissolved in chlorobenzene and stored in vacuum desiccator. Then $[NP(OH)_2]_3$ and oleic acid were mixed in equimolar ratio in DMF, followed by the addition of 50 mL of conc. H_2SO_4 , and refluxed for 6 h. The produced mass was separated, washed with DMF, EtOH and Et_2O successively, dried and stored *in vacuo*.

Qualitative and quantitative estimations for P, N, C, H and O were done as

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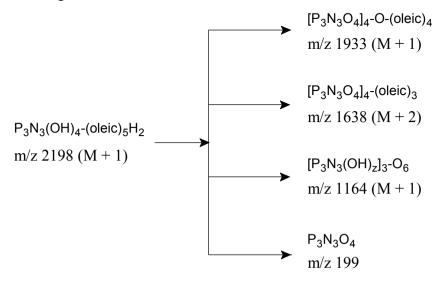
diseribed⁷ by C, H, N Analyser. Mass and I. R. spectra were recorded on Jeol SX-102 (FAB) and Shimadzu 8201-P.C.(400-4000 cm⁻¹, KBr) spectrometers, respectively.

RESULTS AND DISCUSSION

The chemical data for the reaction product of $[NP(OH)_2]_3$ and oleic acid - % found (cal.), 16.81 (16.80), N 7.59 (7.588), C 48.80 (48.78), O 18.80 (18.79), H 8.18 (8.175) and mol. wt. 2212.4 (2214.0) g mol⁻¹, lead us to assign it the structure as $[P_3H_3(OH)_4]_4$ -{O-O-(CH₂)₇-CH=CH(CH₂)₈}₅H₂, which is supported by the mass peak at (m/z) 2198 (M + 1). 17 unit mass less for one OH group than its mol. wt. 2214.0 g/mol. Corresponding to the fragment is given as –

(where-O-C-(CH_2)₇-/CH=CH-(CH_2)₇- CH_2 = oleic group)

Mass of fragments line at various m/z are -



The fragmentation of the product, having prominent peak at m/z 2198 (M+1), into fragments according to the peak line of high intensity (fig-1) may be expressed as follow.

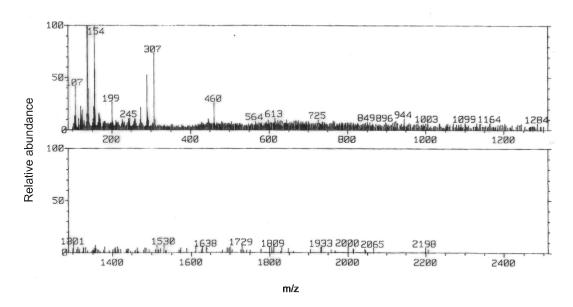


Fig. 1: Mass spectrum of THTPPO

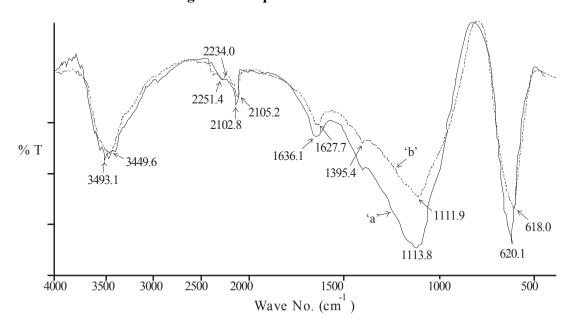


Fig. 2: I. R. spectrum of (a) Ligand and (b) THTPPO

Fig. 3: Structure of THTPPO

The presence of various groups was confirmed by the comparison of I. R. spectra (Fig. 2) of the [N₃P₃(OH)₄]₄-[O-CO-CH₂)₇-CH=CH-(CH₂)₈]₅H₂, which possess some condensed and broad bands at lower frequencies than that of the [NP(OH)₂]₃, the trimer, while a few vibrations have shifted to the higher region. The presence of band in I.R. of product at 618.0 (s), 1627.7 (s) and 2105.2 (s) cm⁻¹ are due to P-N and P=N bonds⁸. A broad band at 1111.9 cm⁻¹ (condensed) for P-OH and at 2251.4 (triplet) cm⁻¹ are due to P-OH bond, with slight change; thus, suggesting the presences of unbonded P-N ring during the reaction of [NP (OH)₂]₃ and oleic acid. This is also supported by the reduction in intensity of N-P-OH band present at 3449.6 (b) cm⁻¹. A new vibrational band in I. R. spectrum of product was observed at 1395.4 (b.w) cm⁻¹, which has be corresponding to HC=CH (conjugated) to alkyl group with P-N ring. It indicator the formation of product, as tetrahydroxy tetraphosphazene penta oleiate, THTPPO, [N₃P₃(OH)₄]₄-(O-CO-(CH₂)₇-CH=CH-(CH₂)₈]₅H₂ as expressed in its polymeric structure, (Fig. 3), which is formed by the elimination H₂O during the reaction of [NP(OH)₄]₃ and oleic acid in presence of conc. H₂SO₄ in the following condensation process shown below –

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