



SPECTRAL CHARACTERIZATION OF REACTION PRODUCT OF OLEIC ACID AND HEXAHYDROXYCYCLOPHOSPHOZENE

ILLA RANI and S. P. S. JADON*

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

ABSTRACT

[NP(OH)₂]₃ was mixed with oleic and refluxed in DMF in presence of conc. H₂SO₄. The mass, produced, was estimated quantitatively, along with its mass and I. R. spectra. The product has been assigned the structure [P₃N₃ (OH)₄]₄-[O-CO-(CH₂)₇-CH=CH-(CH₂)₈]₅H₂ 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, (NPCI₂)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 (NPCI₂)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. H₂SO₄, and refluxed for 6 h. The produced mass was separated, washed with DMF, EtOH and Et₂O successively, dried and stored *in vacuo*.

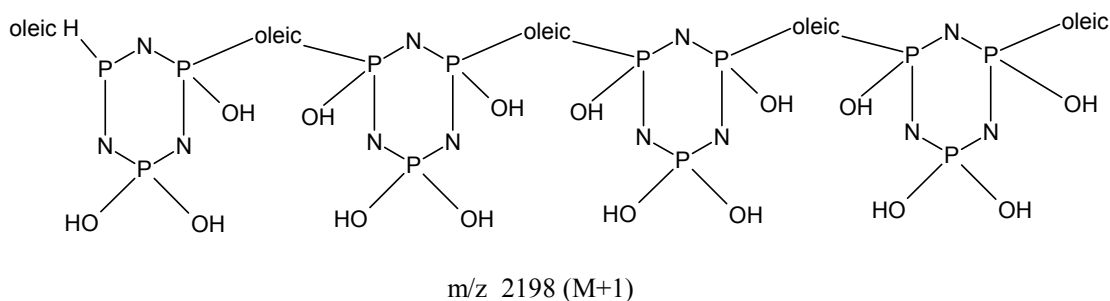
Qualitative and quantitative estimations for P, N, C, H and O were done as

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

described⁷ 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.

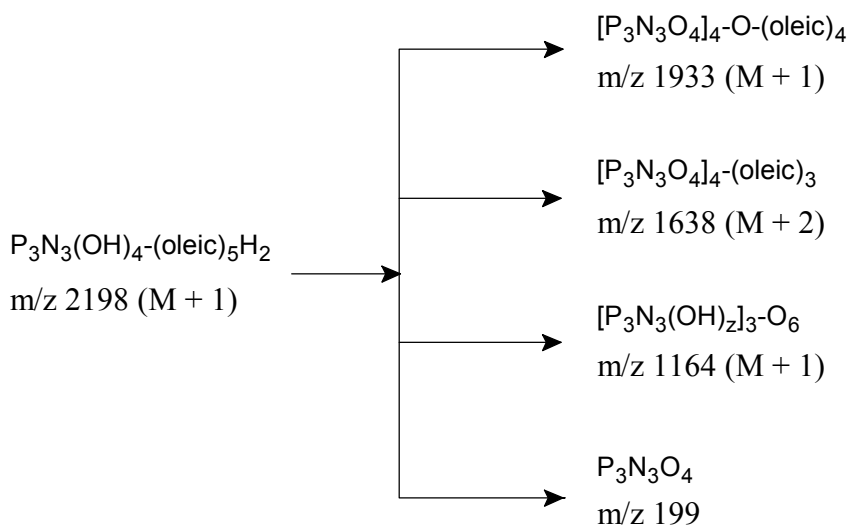
RESULTS AND DISCUSSION

The chemical data for the reaction product of $[\text{NP}(\text{OH})_2]_3$ 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 $[\text{P}_3\text{H}_3(\text{OH})_4]_4\text{-}\{\text{O-O}-(\text{CH}_2)_7\text{-CH=CH}(\text{CH}_2)_8\}_5\text{H}_2$, 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 –

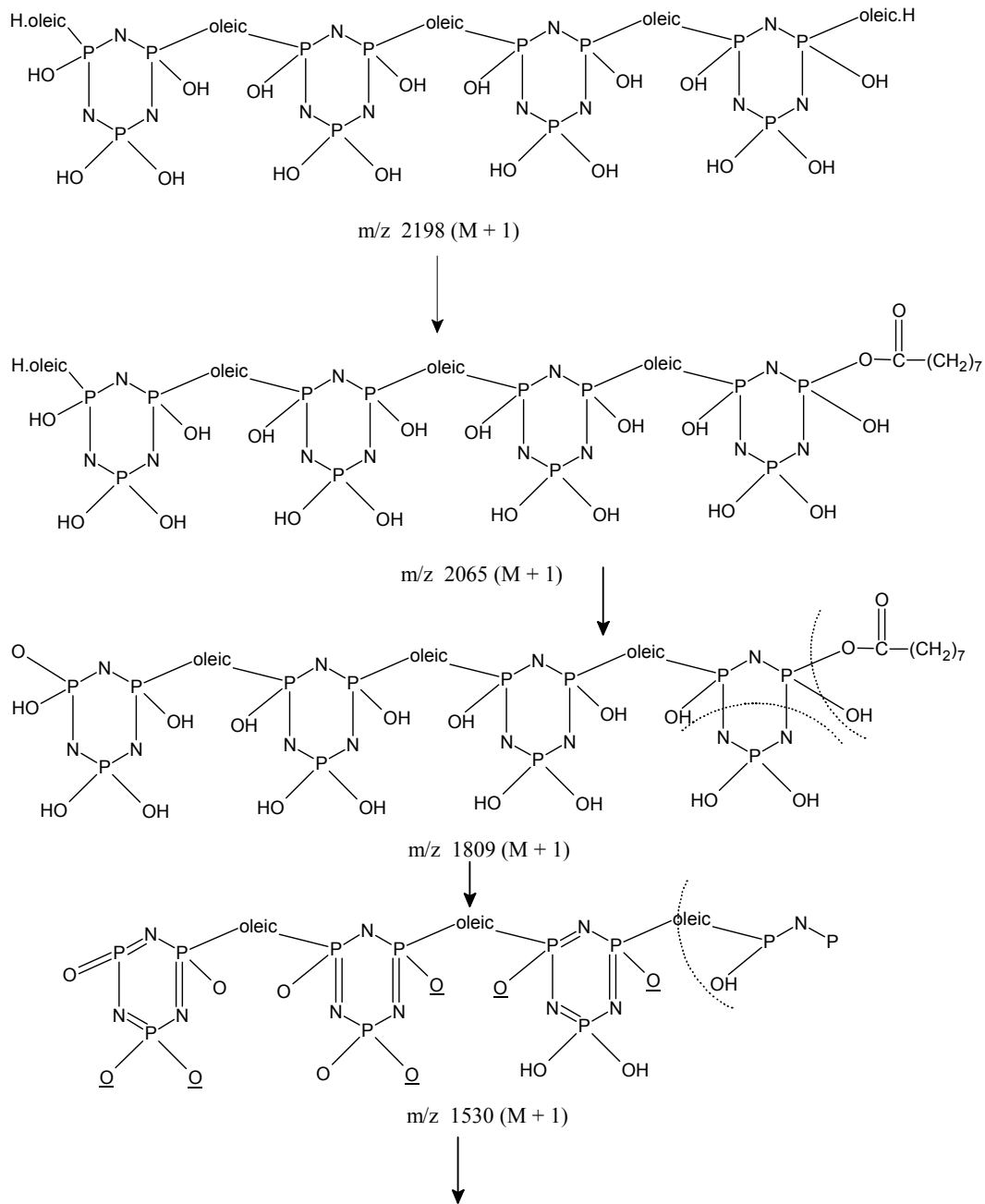


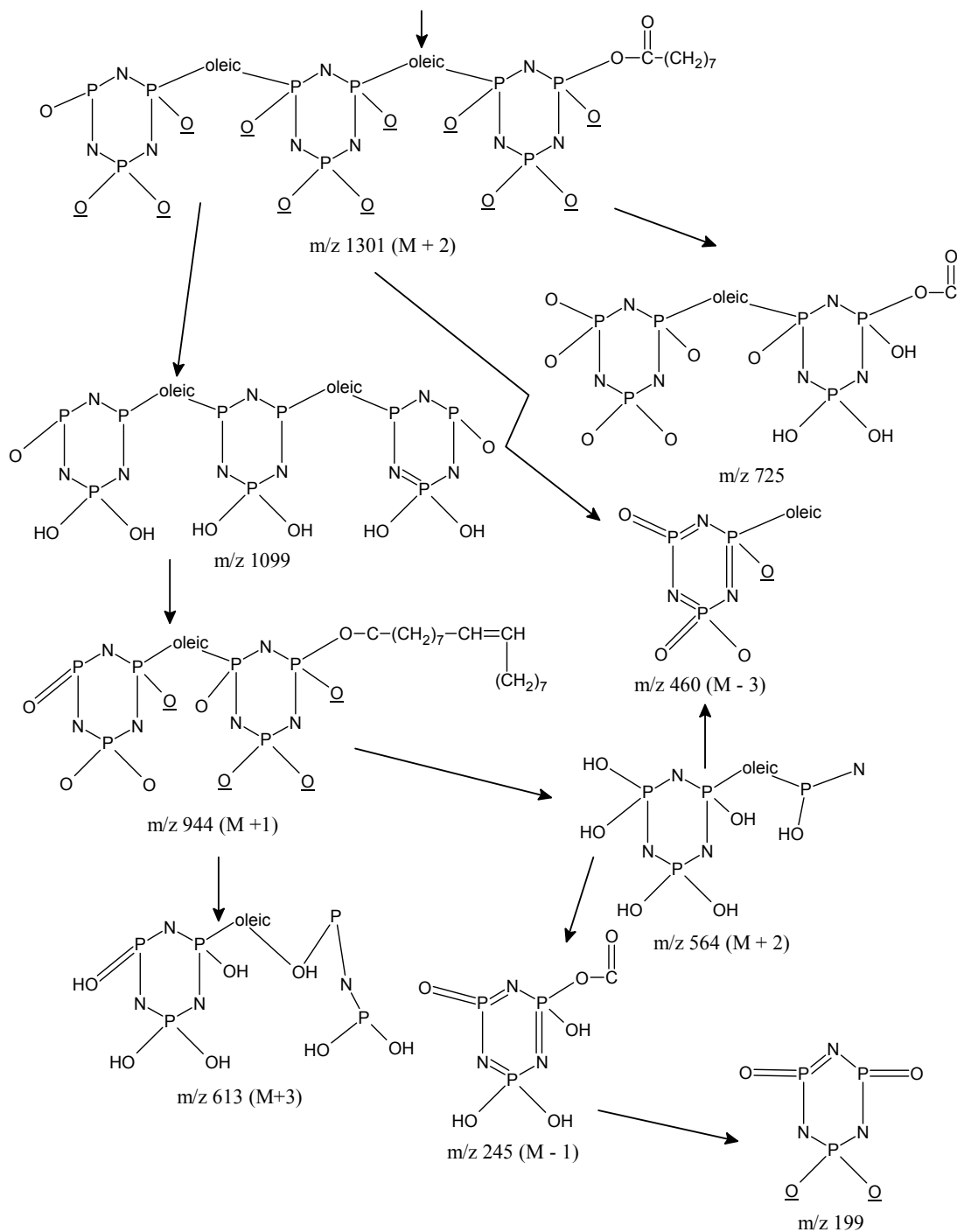
(where $\text{-O-C}-(\text{CH}_2)_7\text{-}/\text{CH=CH}-(\text{CH}_2)_7\text{-CH}_2 = \text{oleic group}$)

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.





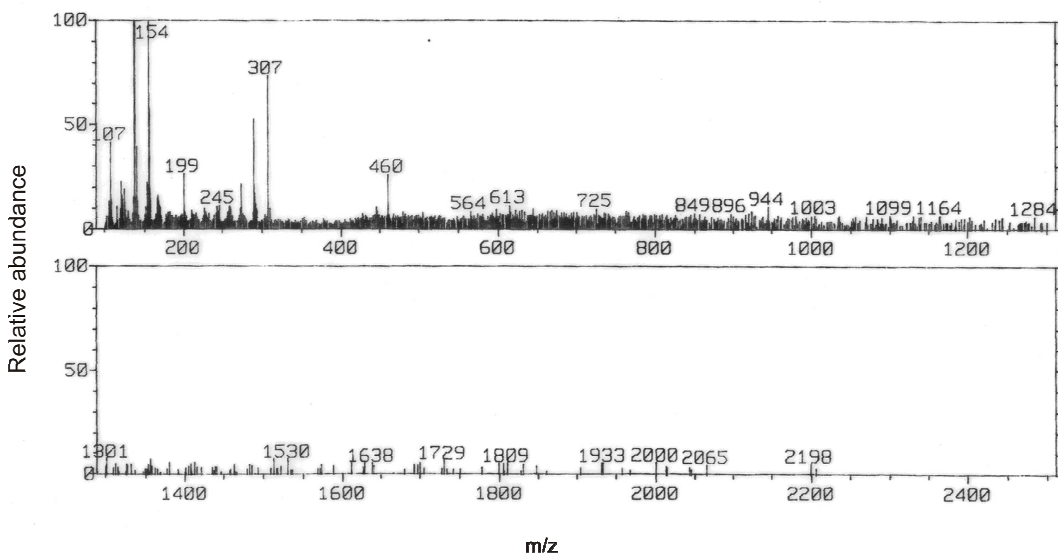


Fig. 1: Mass spectrum of THTPPO

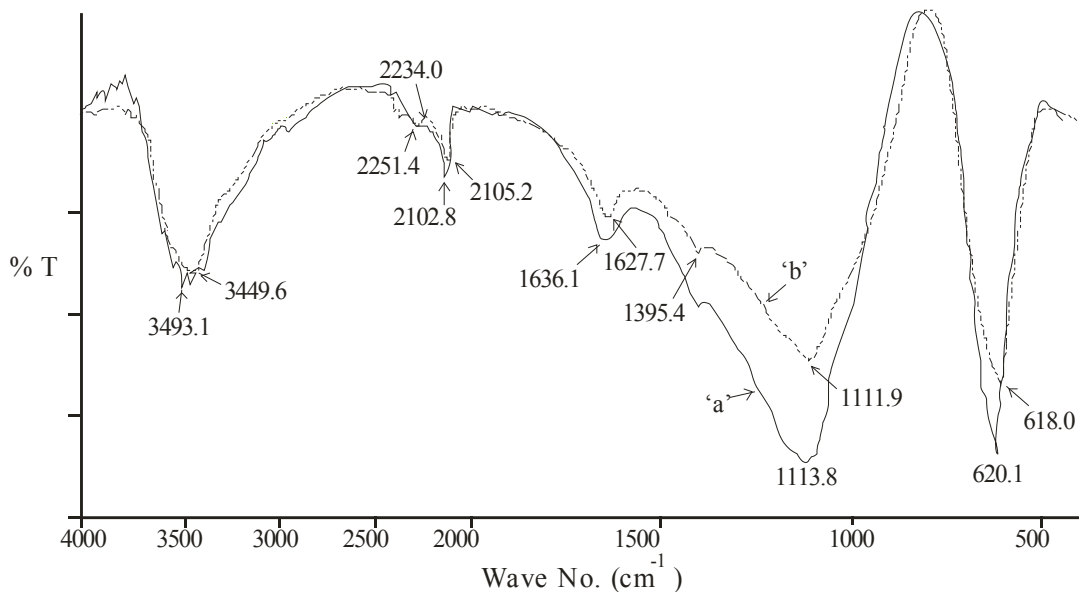


Fig. 2: I. R. spectrum of (a) Ligand and (b) THTPPO

ACKNOWLEDGEMENT

Authors are indebted to Prof. K. P. Modhusudan, Director, C.D.R.I., Lucknow for providing instrumental facilities.

REFERENCES

1. A. Sundermann and W. W. Scholler, *Inorg. Chem.*, **38**, 6261 (1999).
2. Y. P. Singh and S. P. S. Jodon *Asian. J. Chem.*, **19 (4)**, 3280 (2007).
3. N. Jain and S. P. S. Jodon *Asian. J. Chem.*, **18 (1)**, 730 (2006).
4. N. Jain and S. P. S. Jodon *Int. J. Chem. Sci.*, **4**, 285 (2006).
5. Y. P. Singh and S. P. S. Jodon, *Int. J. Chem. Sci.*, **5(2)**, 807 (2007).
6. J. Emsley and P. B. Udy., *J. Chem. Soc. (A)*, 3005 (1970).
7. A. I. Vogel, *A Text book of Quantitative Inorganic Analysis*, E. L. B. S., London, (1968).
8. K. Nakamoto, *The Infra Red and Raman Spectra of Inorganic and Coordination Compounds*, John Wiley Inc. New York. (1978).
9. D. H. Williams and I. Fleming, *Spectroscopic Methods in Organic Chemistry*, Tata McGraw-Hill New York (2004).

Accepted : 09.03.2008