



MASS AND ^1H NMR SPECTRA OF THE COMPLEX OF N-THIOTRITHIAZYL-o-THIOTRITHIAZYL PHENYLALANINATE WITH Zn (II)

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ABSTRACT

The complex of Zn (II) with new ligand N-thiotrithiazyl-o-thiotrithiazyl phenylalaninate was prepared, and characterized by Mass, I.R. and ^1H NMR spectral studies. The complex was found, having molecular formula ZnL_2 . On the basis of IR, ^1H NMR and mass spectra, it has been proved that the complex is quadridentately coordinated.

Key words: Mass, NMR, Zinc, Phenylalaninate, Thiotrithiazyl.

INTRODUCTION

$\text{S}_4\text{N}_3\text{Cl}$ and its complexes have been used as chemical agents for microbials¹⁻³. N-thiotrithiazyl-o-thiotrithiazyl phenylalaninate (N-TTA-o-TTAPA) is obtained by the condensation of thiotrithiazyl chloride and phenylalanine, which formed number of complexes with metal ions, organic as well as inorganic compounds⁴⁻⁹. The investigation of the complex, Zn (II)-bis-N-thiotrithiazyl phenylalaninate are being reported, herewith.

EXPERIMENTAL

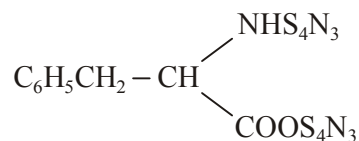
Materials and methods

Phenylalanine, DMF and zinc oxide (B.D.H.) were used, $\text{S}_4\text{N}_3\text{Cl}$ was prepared as reported earlier¹⁰. The new ligand, N-TTA-o-TTAPA was obtained by refluxing $\text{S}_4\text{N}_3\text{Cl}$ and phenylalanine in DMF for 6 h.

The light yellow product obtained, was filtered, washed subsequently with DMF, ether and alcohol; dried and stored in vacuum desiccator.

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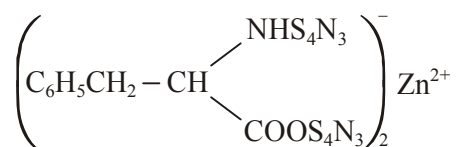
On the basis of quantitative estimation, mass, IR and ^1H NMR spectra, the new ligand has been formulated as –



Syntheis of Zn (II) complex

1 mole of zinc oxide was dissolved in DMF and to this, 2 mol of ligands was added with constant stirring. This mixture was refluxed for 6 h. The light yellow powdered mass obtained, was subsequently washed with ether, dried and stored in vacuum desiccator.

M.P. > 300°C yield (65 %). The chemical data %; found (cal.) Zn; 8.57 (8.60), S 35.00 (35.20), N; 15.22 (15.33), C; 29.44 (29.54), H 2.40 (2.46) and mol. wt. (730.5) 731 g/mol. The complex has been assigned structure as –



Mass, ^1H NMR spectrum and IR spectrum were recorded on Jeol SX-102 (FAB) spectrometer, Bruker DRX-300 spectrometer and Shimadzu 8201 PC IR Hitachi spectrophotometer, respectively. Elemental analysis were obtained from Perkin-Elmer CHN microanalyzer.

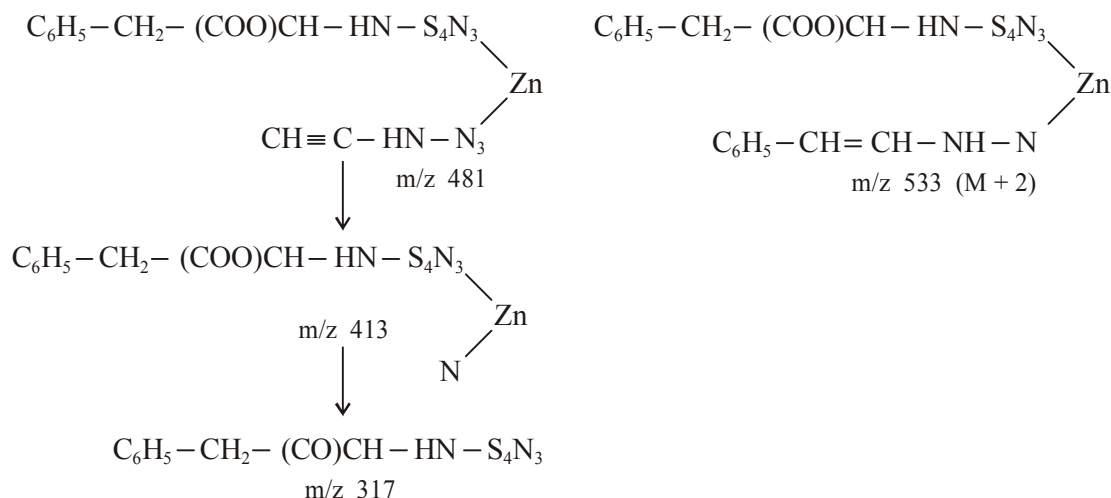
Test for chloride was performed and it was found to be negative.

RESULTS AND DISCUSSION

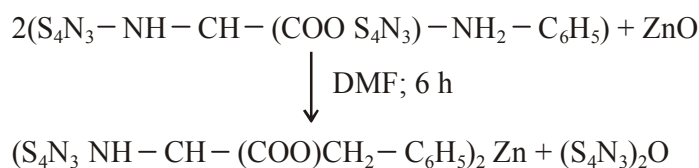
Analytical data show that zinc has formed the 1 : 2 adduct with N-TTA-o-TTAPA. The complex obtained was stable at room temperature.

Mass spectrum

As an additional support, the recorded FAB mass spectrum of the complex shows the estimated molecular weight to be 731.5 g/mol for Zn (II) complex, which is in good agreement with the experimentally determind molecular weight (730.5) of the complex.



Thus, the mass lines for various fragments explained the ZnL_2 complexes formation. The reaction between N-TTA-o-TTAPA and ZnO may be shown as –



In the IR spectrum (Table 1) of the complex, the bands were observed at 465 (d, w), 617 (d, b), 1119, 1397, 1625 and 2359 cm^{-1} , are for the $\text{O} \rightarrow \text{M}$, $\text{N} \rightarrow \text{M}$, new bonding N-S, S-S, S-N bonds of S_4N_3 rings, NH and CO groups, respectively^{11,12}.

The shifting of S-N free band toward lower frequency, suggests the coordination of S_4N_3 ring through nitrogen atom, Lowering of the frequency of COO- groups also confirms its coordination to Zn (II) metal ion. Thus, the ligand, N-TTA-o-TTAPA has quadridentatively coordinated to Zn (II) through the oxygen atom of amino acid and N atom of S_4N_3 ring.

^1H NMR spectrum was recorded and it is interpreted on the basis of available literature. The two sets of triplet signals at chemical shift, δ 0.00 to 1.237 and δ 6.908 to 7.325 ppm are due to the two S_4N_3 rings present on opposite side along with the two sets of multiplet of signals in the range of chemical shift, δ 2.502 – 3.320 ppm for the two phenylalaninate groups.

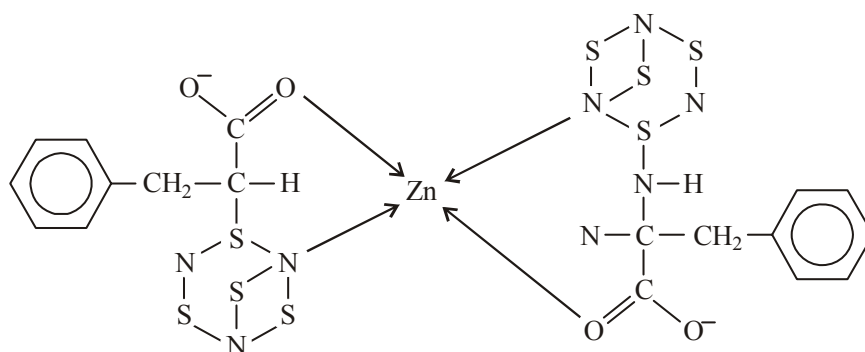
Table 1: IR spectral data of complex

Vibrations	Band assigned	Free constant $K \times 10^5$ (dyn/cm)
465 (d, b)	O \rightarrow M	10.77
617 (d, b)	N \rightarrow M	18.96
1119 (sh)	N – S	18.96
1396 (sh)	S – N in S ₄ N ₃ ring	62.37
1625 (b)	ν (C = O)	97.27
23.59 (sh)	ν (N – H)	277.18

The peaks at chemical shift δ 6.908 – 7.076 are due to excessive interaction of groups with CH₂ groups. The two triplets with chemical shift 7.206 – 7.408 are due to the two CH-CH₂ groups with in the complex. Two signals in the spectrum at chemical shift δ 7.907 and δ 7.964 are due to two phenyl groups present in the complex, while the two signals at chemical shift δ 8.344 and δ 8.372 are on account of two NH groups present in the complex.

The chemical shift change in the ¹H NMR spectrum of the complex also suggest the complex formation.

These data suggest the following structure of the complex (Fig. 1).

**Fig. 1: Structure of complex Zn (II) Bis (N-TTA-o-TTAPA)**

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Revised : 15.03.2010

Accepted : 18.03.2010