



SYNTHESIS AND CHARACTERISATION OF NOVEL ADDUCT OF THIOTRITHIAZYLCHLORIDE WITH AMINO ACID TYROSINE

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Abstract

Thiotrithiazylchloride is a versatile adduct of thiotrithiazyl and chloride ions due to presence of donor atoms and it reacts with metals and as well as organic compounds such as triphenylphosphine and primary amines. In this present work, the novel adduct of thiotrithiazylchloride with amino acid tyrosine has been prepared and its structure established on the basis of mass, IR, EPR and X-rays studies.

Keywords : Thiotrithiazylchloride, Tyrosine, Adduct and Spectral studies.

Received 16.05.2023

Revised 01.08.2023

Accepted 24.09.2023

Introduction

The halogenated derivatives of sulphur nitride have been reported and their complexes with transition metal as well as organic compounds have prepared. However an adduct of thiotrithiazylchloride with amino acid tyrosine was neither prepared, nor used as ligand.

Experimental- All the chemicals used for the synthesis of adduct of thiotrithiazylchloride with amino acid tyrosine were of analytical grade. The used solvents in the synthesis of adduct were purified by standard methods. The elemental analysis was carried out using CHN micro-analyser and also gravimetrically methods. Mass spectrum was recorded on Jeol-SX-102 (FAB) mass spectrometer. The UV and IR were recorded on Perkin-Elmer Lambda 15 UV/ VIS spectrophotometer and Shimadzu 8201 PC IR Hitachi spectrophotometer respectively. NMR spectrum was recorded on Bruker DRX-300 NMR spectrometer.

XRD Spectra were carried out on Philips PW 3710 spectrometers using $\text{Cu}\alpha$ as a source of radiation ($\lambda=1.5405\text{\AA}$) in 2θ range 20-90. The analysis was carried out from S.A.I.F.R.T.M., Nagpur.

Preparation of the novel adduct –Tetrasulfur tetranitride and thiotrithiazylchloride were prepared by known procedures. Thiotrithiazylchloride and amino acid tyrosine was dissolved separately in DMF, after mixing the both solution in refluxing flask, the mixture was refluxed 8 hrs. The resulting product was filtered, washed with alcohol and ether kept in vacuum [MP> 300 C, % yield 55]. Mol.Wt.702.50.C, 30.76 (30.94), H 2.80 (2.86), N 15.45 (16.06), S 34.46 (36.67).

Results and Discussion

Amino acids are the monomers of proteins. The nitrogen atom of NH_2 group of amino acid is the nucleophile, so that S_4N_3^+ ion attached with the N -terminal of amino acid and form the novel adduct. The IR spectrum of the ligand shows bands at 617, 668, 1119 and 1402 cm^{-1} assigned to S – N, S – S, S – N, S_4N_3 ring. In the new ligand the band at 670

cm^{-1} bifurcated into two bands at 668 cm^{-1} and 617 cm^{-1} due to linkage S_4N_3 ring to NH group of tyrosine. The band at 1117 and 1402 has got broadened & decreased in intensity showing the bonding of S – N bond of S_4N_3 ring. The bands at 1596 cm^{-1} the COO group at 2359 cm^{-1} due to the NH group in adduct.

E.P.R. spectrum –

The room temperature, powdered EPR spectrum of the ligand yields a broad signal with g value of 1.921. The eff value 1.663 B.M. shows the ligand to be paramagnetic in nature.

^1H NMR spectrum - The ^1H NMR spectrum of adduct in DMSO – d_6 shows a multi-plate signal between 2.268 – 3.571 ppm due to the presence of S_4N_3 NH proton. The triplet signals between 1.093 – 1.991 ppm due to the $-\text{CH}_2-\text{CH}$ and $-\text{CH}_2$ groups. The signals between 7.024 – 7.367 ppm and 7.954 ppm may be assigned subsequently to the $-\text{C}_6\text{H}_4\text{OH}$ and NH groups protons.

XRD spectrum - From the X – ray powder diffraction pattern of adduct, the value of 'd' (\AA), axial distances (a_0 , b_0 , c_0) and axial angles (α , β , γ) have been calculated for the prominent peaks. The values of a_0 , b_0 , c_0 (19.87 \AA , 19.87 \AA , 24.34 \AA) and α , β , γ (90.000, 90.000, 120.000) suggested the hexagonal geometrical arrangement of the atoms with in adduct.

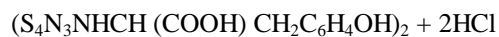
Conclusion

On the basis of mass spectrum, the novel adduct was formulated as

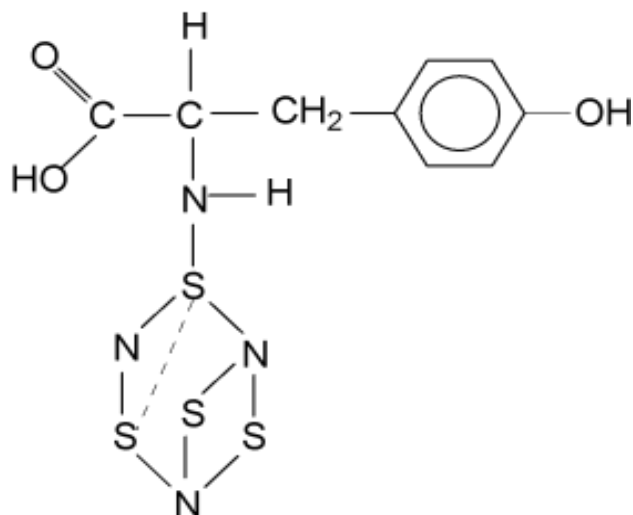
$[\text{S}_4\text{N}_3\text{NHCH}(\text{COO})\text{CH}_2\text{C}_6\text{H}_4\text{OH}]_2$ corresponding to molecular ion peak at $m/z = 698$ suggesting the dimerization of the adduct. The S_4N_3 ring is linked to the nitrogen atom of amino group of the tyrosine molecule by the removal of HCl molecule as confirmed by IR and U.V. spectrum. The novel adduct is paramagnetic in nature and good conductor of electricity with hexagonal geometrical array.

The reaction between $\text{S}_4\text{N}_3\text{Cl}$ and tyrosine amino acid may be shown as:





On the basis of 1H NMR and XRD spectrum the proposed structure of the adduct may be-



Structure of the novel adduct

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