



SPECTRAL INVESTIGATIONS OF Co (II) TETRA SELENIAZIDE COMPLEX

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ABSTRACT

Se₄N₃Br a donor was refluxed with CoCl₂ in DMF for 6-8 h. The brownish black coloured product was obtained, which was analysed qualitatively and quantitatively. The Br⁻ was found absent. The quantitative analysis is also supported by Mass and IR spectra. The complex was assigned as structure (Se₄N₃)₄CoCl₂, where Co has coordinated hexadentately with Se₄N₃ group through its N-atom.

Key words: Hexadentate, Donor, Linkage, Cobalt.

INTRODUCTION

Due to the lone pair of e⁻ in spare on both; Se and N, the coordinated complex of Se₄N₃Br with some metals such as Mn (II) and Fe (III) have been reported^{1,2}. The Mass and I.R. spectral studies of the complex of Se₄N₃Br with Co (II) compound was prepared and result are being presented herewith.

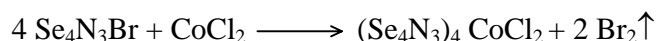
EXPERIMENTAL

Se₄N₃ has been synthesized as reported³⁻⁵ and used as starting material. Se₄N₃Br was prepared by the reaction of HBr on Se₄N₄ (loc.cite). The AR grade chemicals were used through out the work. Se₄N₃Br was refluxed with CoCl₂ (1 : 1) in DMF for 6-8 h. Brownish black coloured product was produced which was separated, filtered and washed subsequently with DMF, alcohol, ether, dried and stored in vacuum desiccator. Qualitative and quantitative estimations were done and mol. wt. was determined by Rasts method, as described⁶. Mass and I.R. spectra were recorded on Jeol SX-102 (FAB) and Shimadzu 8201 PC (4000-400 cm⁻¹) spectrometers, respectively from CDRI, Lucknow.

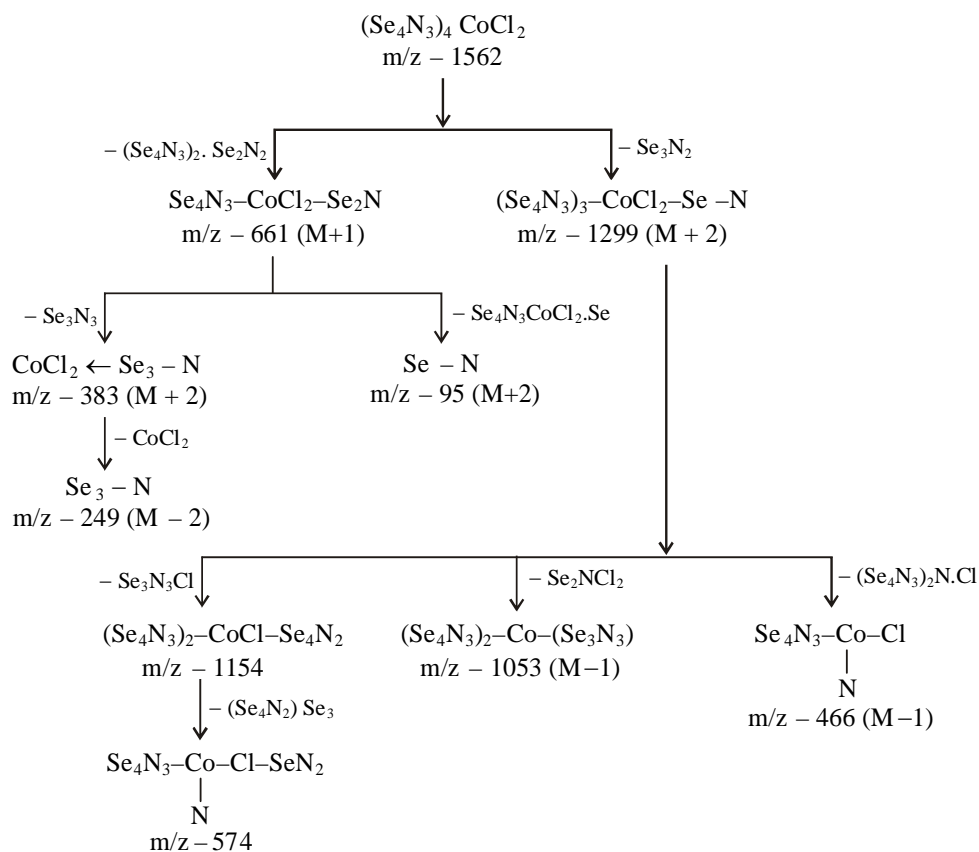
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RESULTS AND DISCUSSION

The complex is brownish black, which is soluble in DMSO and CCl_4 . It decomposes on heating. On the basis of its chemical data and mass spectrogram, i.e.; % Se 80.921 (80.880), N 10.755 (10.749), Co 3.777 (3.775), Cl 4.545 (4.543) and mol. wt. 1562 (1562.8) g/mols it was assigned its mol. formula as $(\text{Se}_4\text{N}_3)_4\text{CoCl}_2$. It indicates that on refluxion of $\text{Se}_4\text{N}_3\text{Br}$ with CoCl_2 , the bromine has been librated and the reaction may be as follow –



This is also supported by the qualitative test for Br^- in complex, which was negative showing the absence of Br in the complexes. While the test for other constituents, Se, Co and N were found positive (loc. cit). The mol. mass of complexes is also supported by the prominent mass peak m/z -1562 in its mass spectrum for the mol. formula $(\text{Se}_4\text{N}_3)_4 \text{CoCl}_2$. The mass pattern for the different mass lines corresponding to the fragments (Table 1) may be explained on the basis of FAB fragmentation process (**Scheme 1**).



Scheme 1

The formation of complex is confirmed by its IR spectrum compared to that of $\text{Se}_4\text{N}_3\text{Br}$ used as donor (Fig. 1, Table 1).

Table 1: Mass and I.R. spectral data of cobalt (II) complex

Mass data		I.R. Spectral data		
m/z	Fragments	Vibrations (cm^{-1})	Bands assigned	Force constant $\text{K} \times 10^5 \text{ dyn cm}^{-2}$
95	Se-N (M+2)	472.3 (s)	$\text{Se-N} \rightarrow \text{Co}$	1.5683
249	$\text{Se}_3\text{-N (M-2)}$	670.8 (b,s)	Se-N-Co	3.1637
311	$\text{Se}_3\text{-N} \rightarrow \text{Co (M+1)}$	1079.4 (b)	$\text{Se-N} \rightarrow \text{Co}$	8.1918
383	$\text{CoCl}_2 \leftarrow \text{Se}_3\text{-N (M+2)}$	1399.4 (quad, b)	$\text{Se-N} \rightarrow \text{Co}$	13.7688
466	$\text{Se}_4\text{-N}_3\text{-Co-Cl (M-1)}$ N	1633.5	Se-N	18.7608
574	$\text{Se}_4\text{N}_3\text{-Co-Cl-Se}_2\text{N}_2$ N	2127.4	Se-N	31.8209
661	$\text{Se}_4\text{N}_3\text{-Co-Cl}_2\text{-Se}_2\text{N (M+1)}$	2259.8	Se-N	35.9050
762	$\text{Se}_4\text{N}_3\text{-Co-Se}_4\text{N}_2 (M+1)$	2360.5	Se-N	39.1762
958	$(\text{Se}_4\text{N}_3)_2\text{-Co-Se}_2\text{N}_2 (M-3)$	3273.3 – 3405.1 (d,b)	$\text{Se-N} \rightarrow \text{Co}$	75.3332 – 81.5220
1053	$(\text{Se}_4\text{N}_3)_2\text{-Co-Se}_3\text{N}_3 (M-1)$			
1154	$(\text{Se}_4\text{N}_3)_2\text{-Co-Cl-Se}_4\text{N}_2$			
1299	$(\text{Se}_4\text{N}_3)_3\text{-CoCl}_2\text{-Se-N (M+2)}$			
1562	$(\text{Se}_4\text{N}_3)_4\text{-CoCl}_2$			

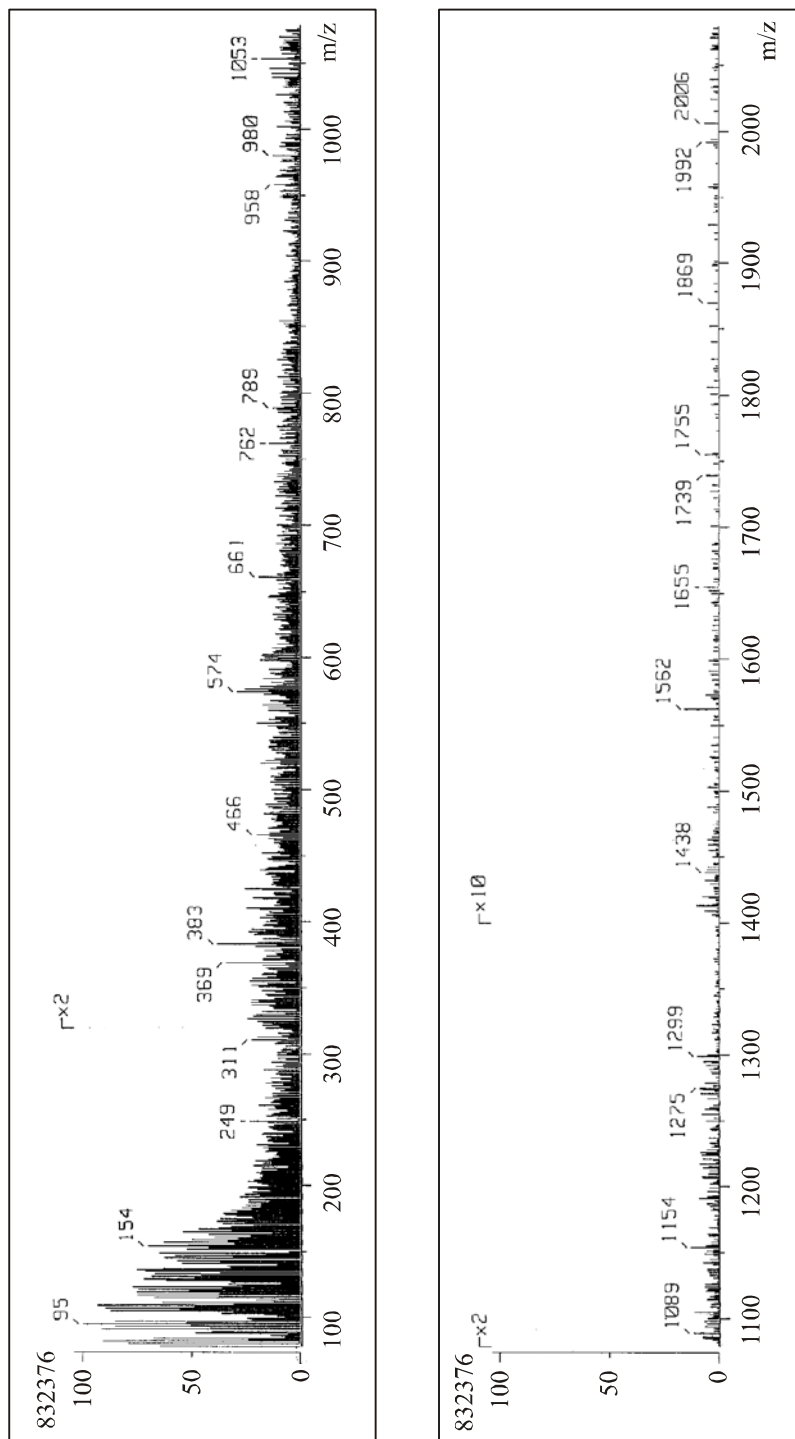


Fig. 1: Mass spectrum of complex

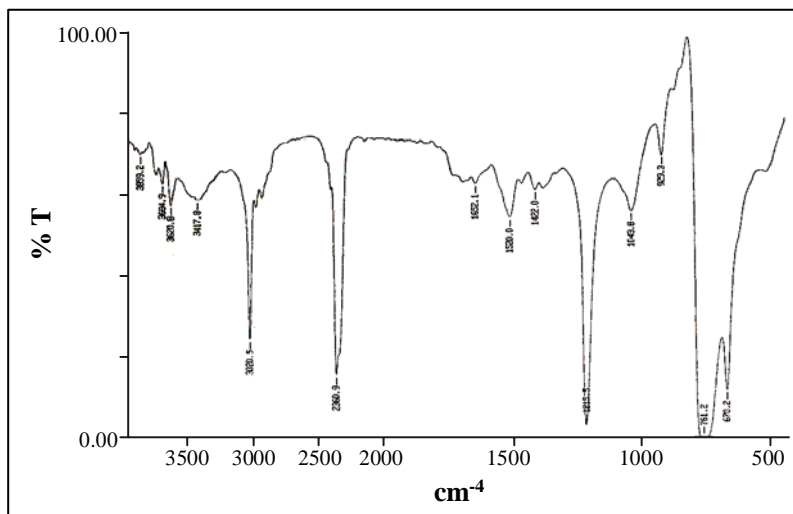


Fig. 2(a): IR Spectrum of ligand

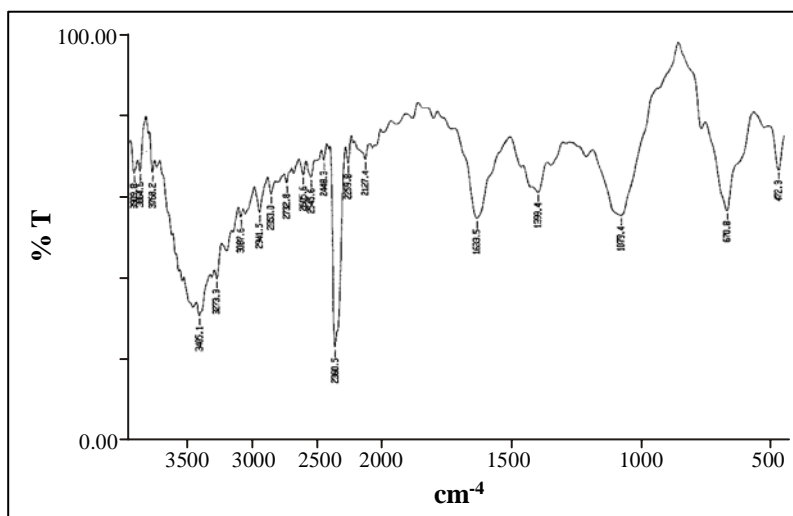


Fig. 2(b): IR Spectrum of complex

The vibrations observed in the I.R. spectrum of ligand at 670.2 and 761.2 cm^{-1} for N-Se-Br have condensed with less intensity to the 670.8 cm^{-1} explaining that Br-Se-N group has coordinated with Co^{2+} with the elimination of Br because a new band 472.3 cm^{-1} in the I.R. spectrum of complex appeared for Se-N \rightarrow Co. Similarly, the sharp peaks at 929.3 and 1043.8 cm^{-1} in the I.R. of ligand have diminished and appeared as a new broad band at 1079.4 cm^{-1} in the higher region of I.R. spectrum of complex suggesting coordination of

Se₄N₃ to Co atom. A broad quardent at 1399.4 cm⁻¹ also indicates the quardant linkage of Se-N bands to Co atom entirely diffrent from other coordinate linkage.

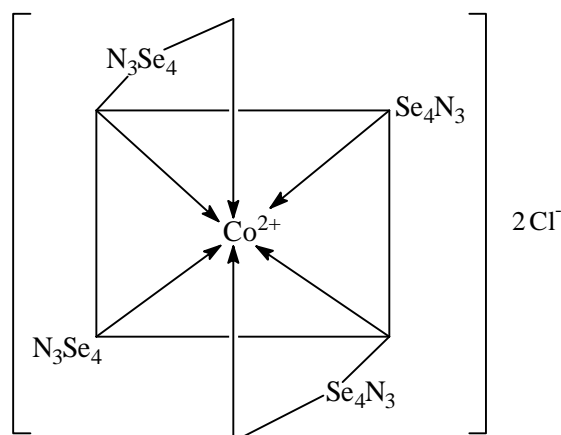


Fig. 3: Structure of the complex (Se₄N₃)₄CoCl₂

The sharp vibration at 2360.5 cm⁻¹ in both; I.R. of ligand and complex is for free Se-N band. In higher region, the peaks appearing at 3020.5 and 3417 cm⁻¹ in the I.R. spectrum of ligand has merged, forming a broad and condensed band at 327.3-3405.1 cm⁻¹ suggesting linkage of the Se₄N₃ group to Co²⁺. The value of force constant calculated⁷ for the band also supports the linkage of Se₄N₃⁺ to Co (II) ion from the I.R. spectrum of complex. It is inferred, that Se₄N₃ ions have hexadentedly coordinated to the Co atom in two different ways as shown by its structure forming octahedral geometry (Fig. 2).

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