



CHARACTERIZATION OF THE COMPLEX OF $\text{Se}_4\text{N}_3\text{Br}$ WITH Fe (III): MASS & I. R. SPECTRA

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ABSTRACT

$\text{Se}_4\text{N}_3\text{Br}$ was refluxed with FeCl_3 in DMF and the product formed, was analyzed quantitatively as well as Mass and I.R. Spectrometrically and assigned as: $(\text{Se}_4\text{N}_3\text{Br})_3.\text{FeCl}_3$.

Key words: $\text{Se}_4\text{N}_3\text{Br}$, Mass and IR spectra.

INTRODUCTION

The halogenated derivative of Se_4N_4 , such as $\text{Se}_3\text{N}_2\text{Cl}_2$, $\text{Se}_4\text{N}_3\text{Cl}$ etc. have been reported.^{1,2} The only products of $\text{Se}_4\text{N}_3\text{Cl}$ by the reaction of SeCl_4 with urea and thiourea have been synthesized and investigated^{3,4}. The complexes of $\text{Se}_4\text{N}_3\text{Br}$ with metal compounds, have not been studied till now. Hence the complex of $\text{Se}_4\text{N}_3\text{Br}$ with FeCl_3 was synthesized and investigated with the help of its Mass and I.R. spectra.

EXPERIMENTAL

$\text{Se}_4\text{N}_3\text{Br}$ was prepared by the reaction of HBr on Se_4N_4 ^{5,6} which was prepared by ammoniation of SeBr_4 in benzene. $\text{Se}_4\text{N}_3\text{Br}$ and FeCl_3 were mixed in equimolar ratio in DMF and refluxed for 6 h. The yellowish brown coloured mass, formed, was separated by filtration, washed subsequently with DMF, alcohol and ether, dried and stored in vacuum desiccator. 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. The quantitative estimations were done by well known methods⁷. The molecular weight was determine by Rast's method.

RESULTS AND DISCUSSION

The complex is yellowish brown solid soluble in DMSO. On the basis of chemical

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data; % found (Cal.) Se 64.11 (64.21), Br 16.23 (16.26), N 8.52 (8.53), Cl 7.20 (7.21), Fe 3.78 (3.79), and mol. wt. 1478.6 (1478.0) gram/mols, the complex is assigned as $(\text{Se}_4\text{N}_3\text{Br})_3 \cdot \text{FeCl}_3$ which is supported by the mass line, found its Mass spectrum (Fig. 1), at m/z 1478 for the fragment $(\text{Se}_4\text{N}_3\text{Br})_3 \text{FeCl}_3$ ($M + 2$). The other prominent mass lines in its mass pattern may be expounded on the basis of FAB fragmentation process as below:

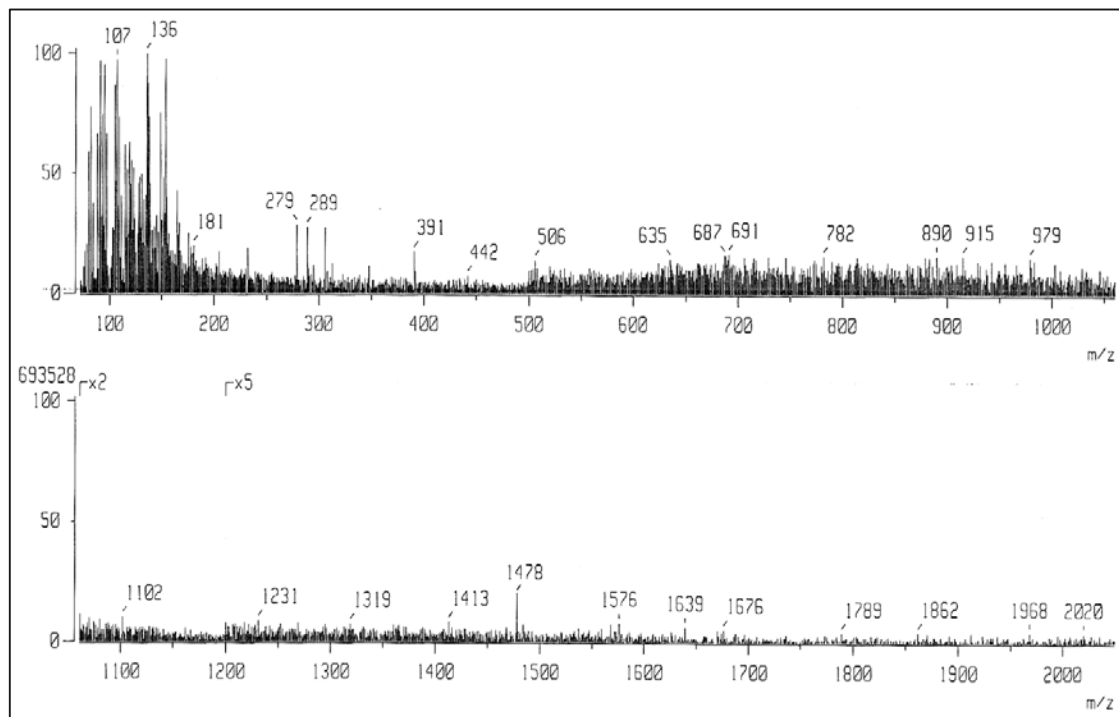


Fig. 1: Mass spectrum of complex

It confirms the molecular formula of the complexes as $(\text{Se}_4\text{N}_3\text{Br})_3 - \text{FeCl}_3$.

The formation of complex is also sustained by the vibrations observed in its IR spectrum compared to that of ligand. The frequencies at 670.2 to 761.2 cm^{-1} , found in the I.R. spectrum (Fig. 2a) of ligand has mixed, shortened and appeared at 669.9 cm^{-1} in IR of the complex (Fig. 2(b)), explaining the coordination of Se-N bond to Fe metal atom i.e. $\text{Se-N} \rightarrow \text{Fe}$. The vibrations observed in the region 929.3 to 1520.0 cm^{-1} in IR of ligand (Fig. 2) has either eliminated or shifted to higher region (Fig. 3) indicating the linkage of $\text{Se}_4\text{N}_3\text{Br}$ to FeCl_3 . Similarly assignment at 1652.1 to 2360.9 cm^{-1} has condensed, showing the confirmation of coordination of Se-N band to Fe atom. The broad band at 3405.9 cm^{-1} has appeared due to lowering the frequency 3417.8 cm^{-1} for Se-N band present in the complex.

From these results it is concluded that $\text{Se}_4\text{N}_3\text{Br}$ has coordinated hexadentately to FeCl_3 as shown by its structure, Fig. 4.

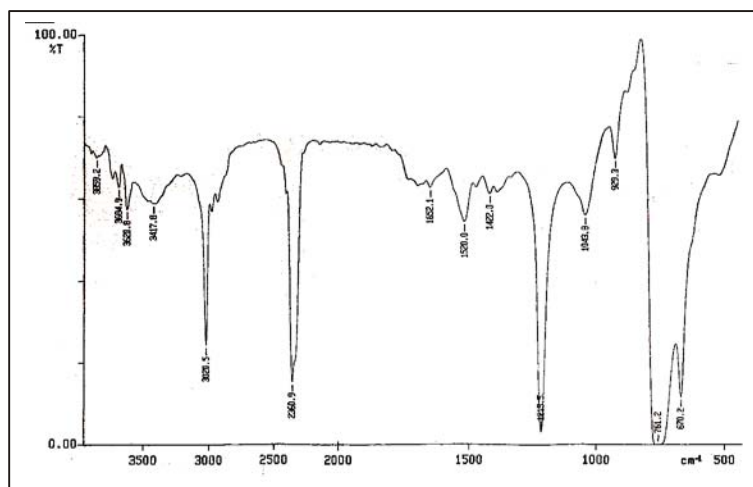
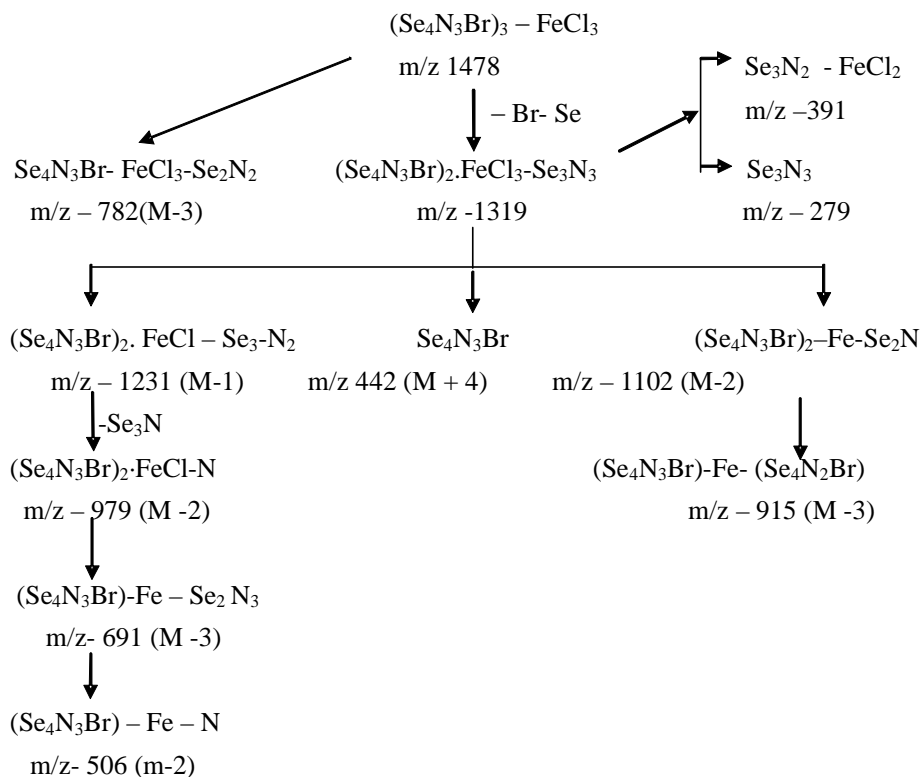


Fig. 2: IR spectrum of ligand

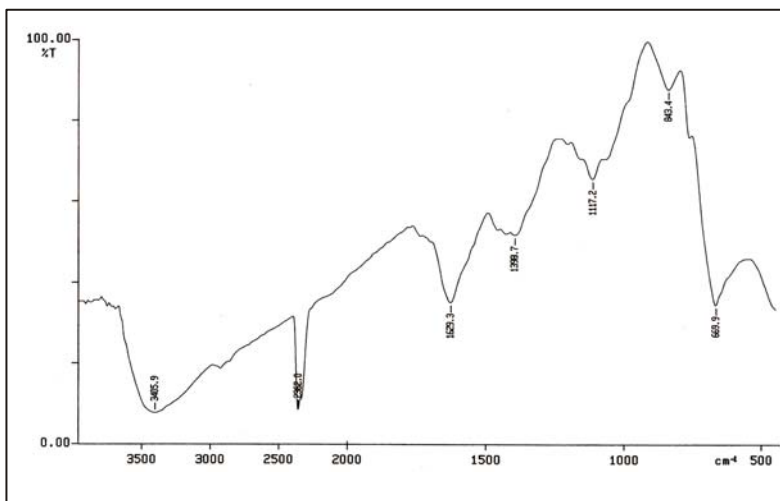


Fig. 3: IR spectrum of complex

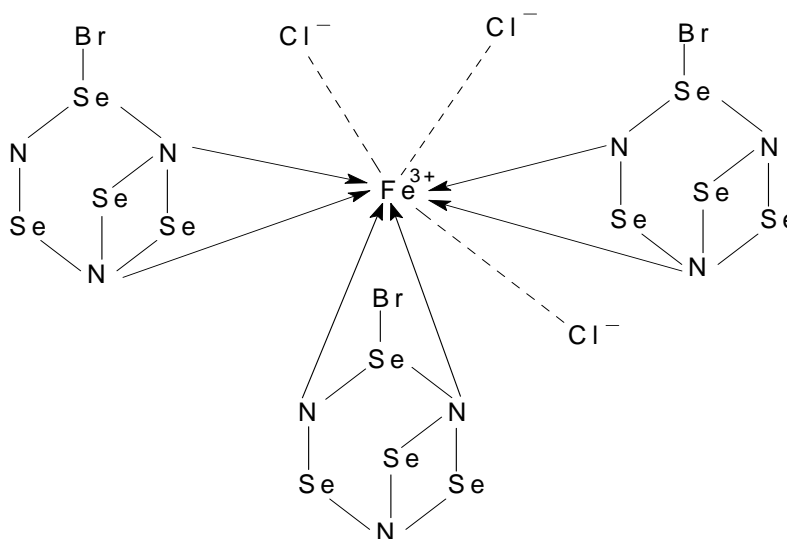


Fig. 4: Structure of the complex hexadentated coordinated with octahedral geometry

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