MASS AND IR SPECTRAL STUDIES OF THE REACTION PRODUCT OF L-LEUCINE AND Se$_4$N$_3$Br
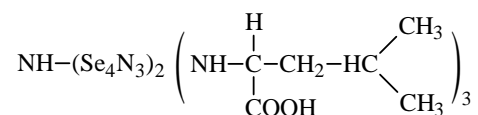
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ABSTRACT

The reaction product of Se$_4$N$_3$Br with L-Leucine, an amino acid was synthesized in dichloroethane. The structure of the product is assigned on the basis of quantitative estimations, mol. wt., mass and IR spectrometric analysis as –

$$NH-(Se_4N_3)_2 \left(NH-\underset{\underset{COOH}{|}}{\overset{\overset{H}{|}}{C}}-CH_2-HC \underset{CH_3}{\overset{CH_3}{<}} \right)_3$$

Key words: Se$_4$N$_3$Br, L-leucine.

INTRODUCTION

The adducts of Se$_4$N$_3$Cl with phenylhydrazine, urea and thio-urea have been reported[1,3], but the adducts of Se$_4$N$_3$Br with amino acids have not been synthesized till now. Therefore, it is intended to prepare the adducts of Se$_4$N$_3$Br with L-leucine and to investigate the reaction product, by mass and IR spectrometrically.

EXPERIMENTAL

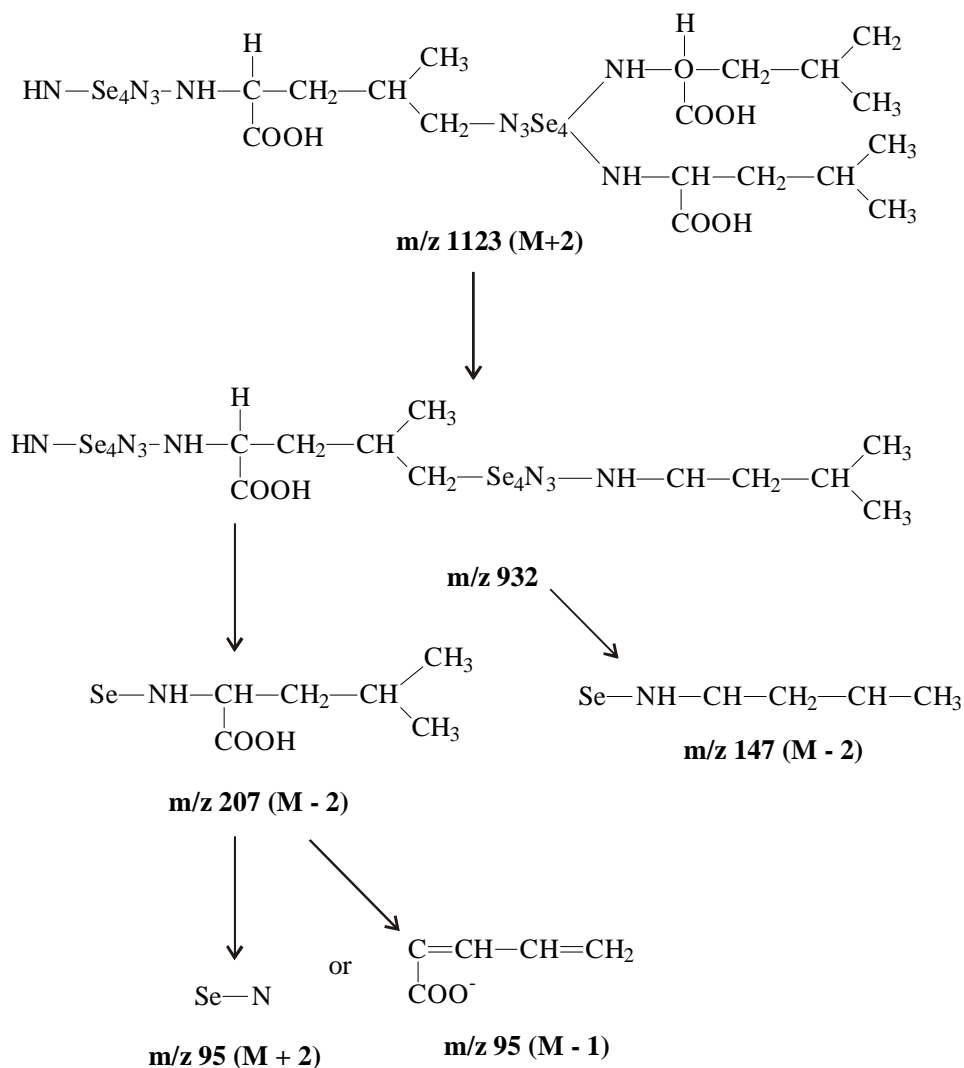Se$_4$N$_3$Br was prepared by the reaction of HBr on Se$_4$N$_4$, which was synthesized as reported[1-7] by the reaction of ammonia on Se$_2$Cl$_2$ at 0°C. The adduct of Se$_4$N$_3$Br with L-Leucine was prepared by refluxing the equimular mixture of both in dichloroethane for 6-8 h. The product, obtained, was separated, washed with dichloro-ethane, alcohol and ether, dried and stored in a vacuum desiccator over fused calcium chloride. The mass and IR spectra of the product were recorded on Jeol SX-102 (FAB) and Perkin Elmer RX1 (450-4000 cm^{-1}) spectrometers, respectively.

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RESULTS AND DISCUSSION

The reaction product is yellowish orange solid, soluble in benzene. On the basis of quantitative estimation, % found (Cal.) Se 56.248 (56.348), N 12.456 (12.489), C 19.223 (19.269), O 8.543 (8.564), H 3.292 (3.301) and molecular weight 1120.6 (1121.0) g/mol. the adduct has been assigned as HN- $(Se_4N_3)_2$-(NH-CH-COOH-CH$_2$-CH $(CH_3)_2)_3$, which is supported by the prominent mass line at m/z 1123 (M+2) in its mass spectrum (Fig. 1).

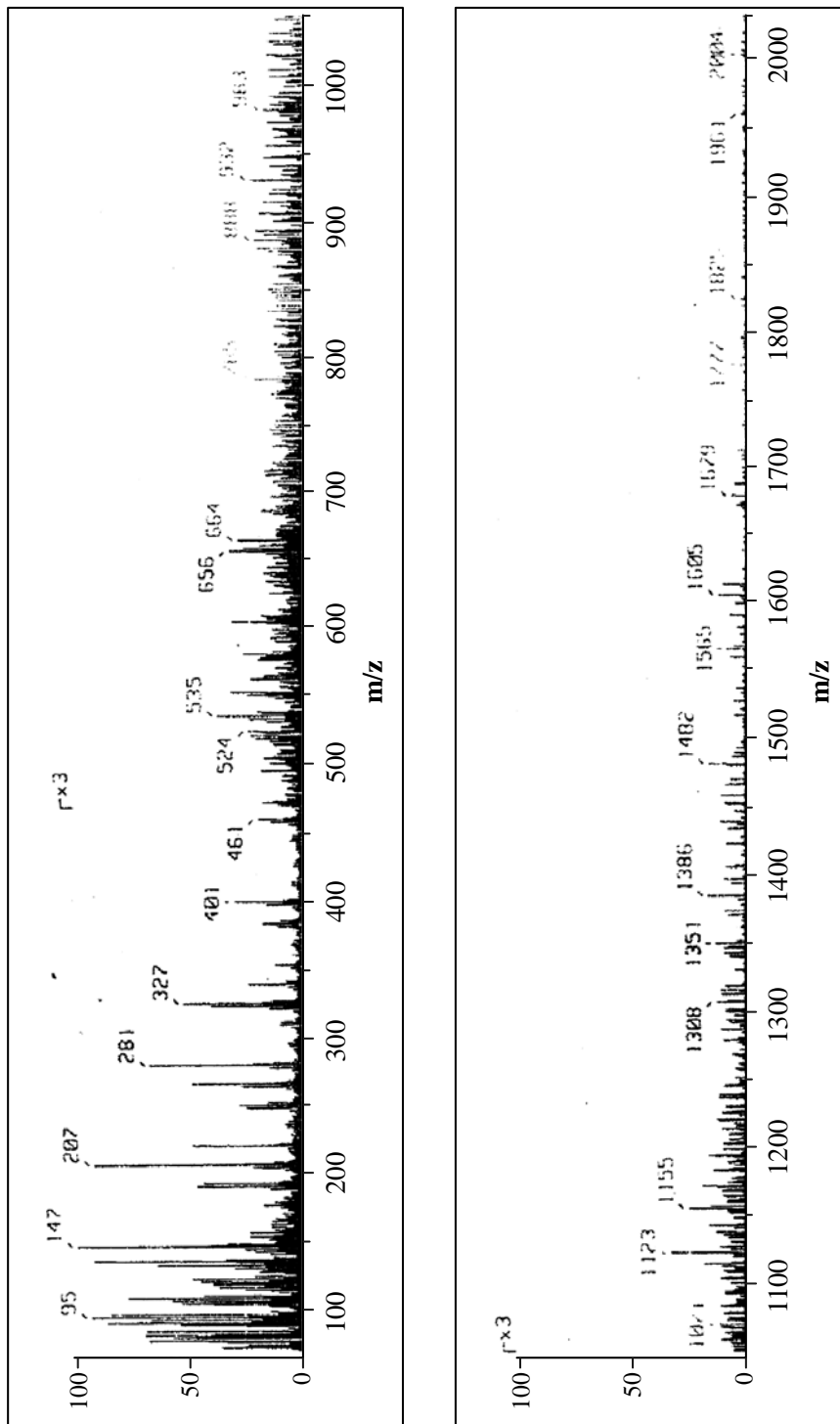The other mass lines in the mass pattern may be explained by FAB fragmentation process as follow:

m/z 1123 (M+2)

m/z 932

m/z 207 (M - 2)

m/z 147 (M - 2)

m/z 95 (M + 2)

m/z 95 (M - 1)

Fig. 1: Mass spectrum of Adduct

From this mass spectrum, it is proved that Se_4N_3Br and L-Leucine has reacted in 1 : 3 ratio according the following reaction.

$$2\ Se_4N_3Br + 4\ NH_2 - CH - CH_2 - CH \begin{cases} CH_3 \\ CH_3 \end{cases} \longrightarrow$$

with $COOH$ below the CH.

$$-HN-(Se_4N_3)_2 - \left(NH-CH-CH_2-CH \begin{cases} CH_3 \\ CH_3 \end{cases} \right)_3$$

with $COOH$ below the CH.

m/z 1123 (M + 2)

$$+\ C=CH-CH=CH_2\ +\ CH_4\ +\ 2HBr +\ 4H_2$$

with COO^- below the C.

m/z 95 (M - 1)

Scheme 1

The formation of the product is also confirmed by the vibration observed in its IR spectrum (Fig. 2, Table 1) compared to that of Se_4N_3Br.

Table 1: I.R. Spectral Data of the adduct

S. No.	Vibrations cm^{-1}		Bands assigned (c)	Force constant K × N. (d)
	Ligand (a)	Product (b)		
1	670.2	543.3	Se-N	2.075
2	761.2	579.8	Se-N	2.363
3	929.8	647.5	Se-N	2.9471
4	1043.8	729.8	Se-N	3.744
5	1215.5	761.5	Se-N	4.077
6	1422.0	803.1 (s)	Se-N	4.534

S. No.	Vibrations cm^{-1}		Bands assigned (c)	Force constant K × N. (d)
	Ligand (a)	Product (b)		
7	1520.0	944.6 (s)	Se-N	6.2735
8	1652.1	940.7 (s)	Se-N	6.22182
9	2360.9	1105.6 (s)	C-O	4.955
10	3020.5	1212.1 (s)	CH$_3$	0.8012
11	3417.8	1400.9 (s)	CH$_3$	1.070
12	3620.8	1524.6	C-C	8.241
13	3684.9	1577.3 (b,d)	Se-N	17.49
14		1703.0 (b,d)	COOH	11.756
15		1956.0 (b,w)	Se-N	26.90
16		2339.5	Se=N	38.4823
17		2362.2	Se=N	39.2327
18		3160.9 (b)	N-H	5.449
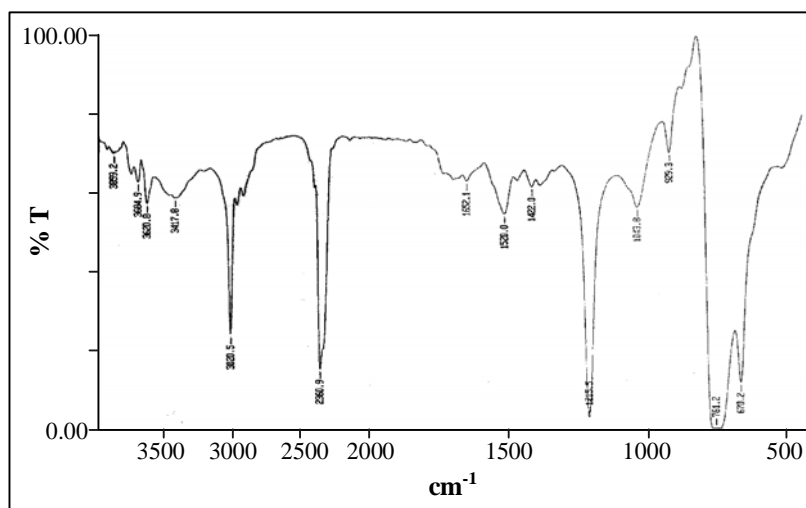
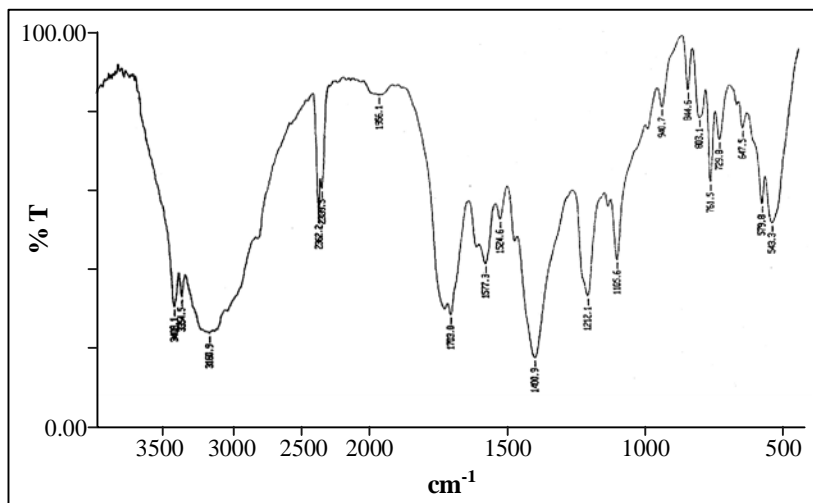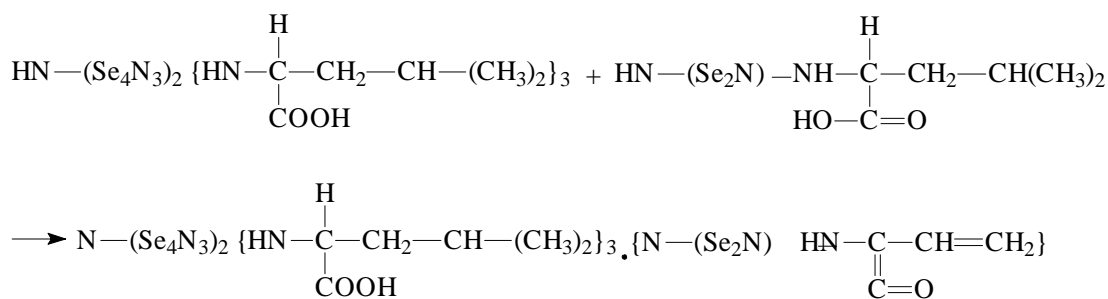**Fig. 2(a): IR Spectrum of Ligand (Se$_4$N$_3$Br)**
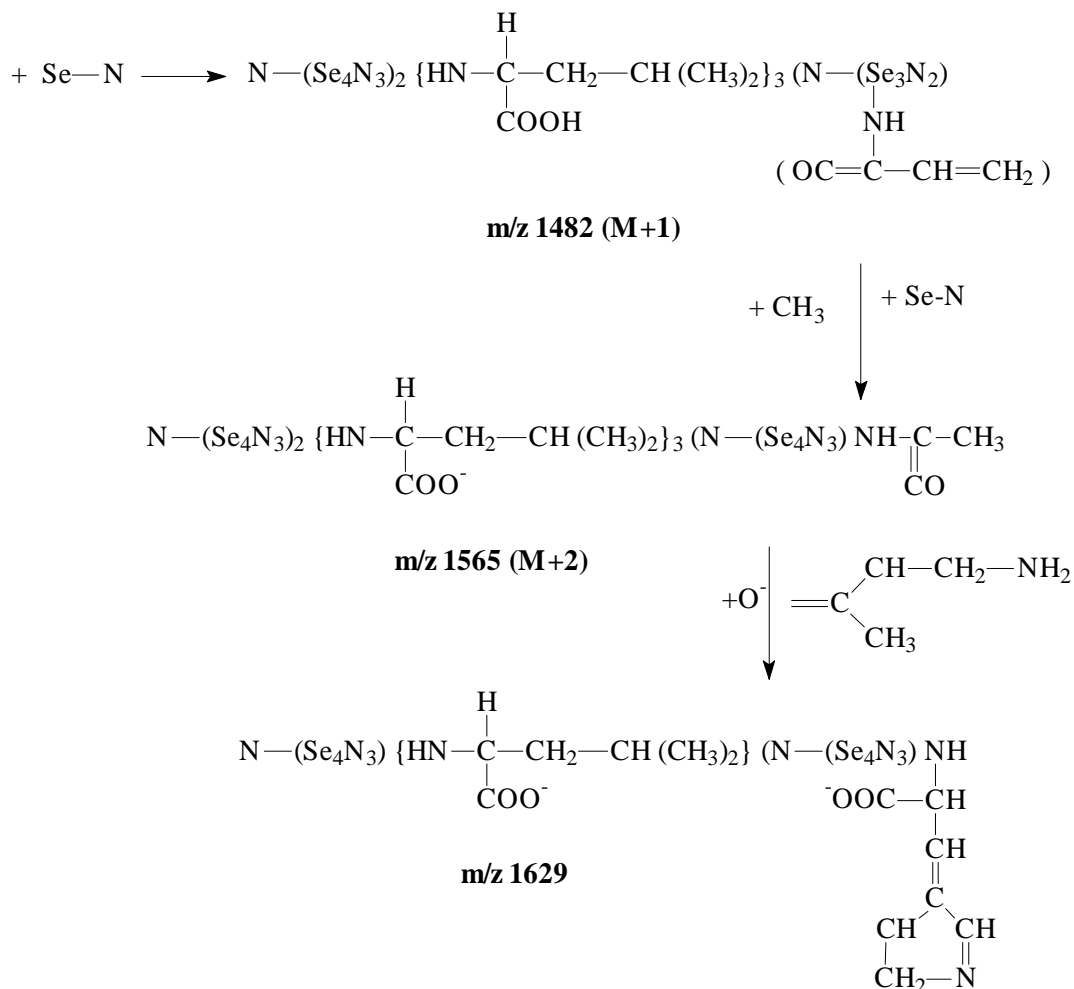
Fig. 2(b): IR Spectrum of Adduct

The appearance of other mass lines at m/z 1386, 1482, 1507 and 1629 in its mass pattern may be impounded on the basis of recombination of Leucine phosphazanide and other fragments formed as follows:

The vibrations appeared in I.R. spectrum (Fig. 2, Table 1) at 543.3 cm^{-1}, 579.8 cm^{-1}, 647.5 cm^{-1}, 729.8 cm^{-1}, 761.5 cm^{-1}, 803.1 cm^{-1}, 944.6 cm^{-1}, 940.7 cm^{-1} are for the Se-N bands while the vibration 1105.6 cm^{-1} for C-O band and 1212.1 cm^{-1}, 1400.9 cm^{-1} for CH$_3$ group, 1524.6 cm^{-1} for C-C band and 1703.0 cm^{-1} for COOH group, 3160.9 cm^{-1} for C-H band and 3354.5 cm^{-1}, 3408.1 cm^{-1} for N-H band, of the L. Leucine amino acid, showing its presence and linkage in the adduct alongwith Se$_4$N$_3^-$ion.

The results revealed that Se$_4$N$_3$Br has reacted with L-Leucine an amino acid with formation of adduct and eliminating HBr during their reaction as mentioned Scheme 1.

HN—(Se$_4$N$_3$)$_2$ {HN—C(H)—CH$_2$—CH—(CH$_3$)$_2$}$_3$ + HN—(Se$_2$N) —NH—C(H)—CH$_2$—CH(CH$_3$)$_2$
                  | |
                COOH   HO—C=O

⟶ N—(Se$_4$N$_3$)$_2$ {HN—C(H)—CH$_2$—CH—(CH$_3$)$_2$}$_3$.{N—(Se$_2$N)   HN—C—CH=CH$_2$}
                  | ||
                COOH   C=O

m/z 1386 (M - 2)

$$+ \ Se\!-\!N \ \longrightarrow \ N\!-\!(Se_4N_3)_2 \ \{HN\!-\!\overset{\overset{\displaystyle H}{|}}{\underset{\underset{\displaystyle COOH}{|}}{C}}\!-\!CH_2\!-\!CH\,(CH_3)_2\}_3 \ (N\!-\!(Se_3N_2)$$

$$\underset{(\ OC\!=\!\overset{|}{C}\!-\!CH\!=\!CH_2\ )}{\overset{NH}{\underset{|}{}}}$$

m/z 1482 (M+1)

$$+ CH_3 \quad\Big|\quad + \ Se\text{-}N$$

$$N\!-\!(Se_4N_3)_2 \ \{HN\!-\!\overset{\overset{\displaystyle H}{|}}{\underset{\underset{\displaystyle COO^-}{|}}{C}}\!-\!CH_2\!-\!CH\,(CH_3)_2\}_3 \ (N\!-\!(Se_4N_3) \ NH\!-\!\overset{}{\underset{\underset{\displaystyle CO}{\|}}{C}}\!-\!CH_3$$

m/z 1565 (M+2)

$$+O^- \quad\Big|\quad \overset{CH\!-\!CH_2\!-\!NH_2}{\underset{CH_3}{=\!C}}$$

$$N\!-\!(Se_4N_3) \ \{HN\!-\!\overset{\overset{\displaystyle H}{|}}{\underset{\underset{\displaystyle COO^-}{|}}{C}}\!-\!CH_2\!-\!CH\,(CH_3)_2\} \ (N\!-\!(Se_4N_3) \ NH$$

$$^-OOC\!-\!CH$$

$$\underset{\underset{CH_2\!-\!N}{\overset{CH}{}}}{\overset{CH}{\underset{C}{}}}$$

m/z 1629

Scheme 1

The presence of Se – N and Se = N bands in Se_4N_3 ring is also inferred by the values of force constants (Table 1), calculated from the frequencies appear in its I.R. spectrum.

ACKNOWLEDGMENT

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