



INVESTIGATIONS OF BROMO Fe (III) TETRA SELENIAZIDE COMPLEX:- U.V., E.P.R. & X.R.D. SPECTRA

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ABSTRACT

To confirm the nature of bonding and geometry of the complex, bromo Fe(III) tetra seleniazide, $(Se_4N_3Br)_3FeCl_3$, its U.V., E.P.R. and X.R.D. spectra, recorded, were analyzed. From the results it is concluded that the complex is a good conductor having paramagnetic character and Triclinic geometry.

KEYWORDS :- Se_4N_3Br , Triclinic, Geometry, Magnetic Susceptibility.

INTRODUCTION

Being the lone pair electron in spare on both Se and N atom, Se_4N_3Br , it was used as ligand and synthesized by the reaction of dry HBr on Se_4N_4 ^{1,2}. The complexes of Se_3N_2Cl and $Se_3N_2Cl_2$ with metals have been investigated^{3,4}. The compounds of Se_4N_3Cl with Urea and Thiourea have also been reported^{5,6}. Se_4N_3Br coordinated with the metal compounds to form their complexes⁷⁻⁹. In continuation the work done, this paper is being presented here with.

EXPERIMENTAL

Se_4N_3Br prepared (loc site.), was mixed with $FeCl_3$ in equimolar ratio in D.M.F. and refluxed for 6hrs till yellowish brown mass was precipitated. The product was filtered, washed successively with D.M.F., alcohol and ether, dried and stored. The complex was tested qualitatively for its constituents elements. Perkin-Elmer-Lambda-15 spectrophotometer (200-800nm) Varian's X-E-4 band(R.T.) and PW-1710 X-ray power diffractometer ($\lambda=1.5418\text{\AA}$, $Cu K_\alpha$ as a source of radiation) in the 2θ range $0^\circ-80^\circ$ were used to record their U.V., E.P.R. and X.R.D. spectra respectively. The complex is also

used against *S. aureus* (gram +ve) and *E. coli* (gram -ve) bacteria to check their inhibitions.

RESULTS AND DISCUSSION

For the molecular formula, $(\text{Se}_4\text{N}_3\text{Br})_3\text{FeCl}_3$, of the complex, mol.wt. 1478.6 gm/mol. found, is supported by the mass line m/z in its mass spectrum (loc. site.).

U.V.Spectra:

The electronic spectrum of the complex possesses three bands at 200, 237.5, 285.0 nm (fig-1). The bands at 200 and 237.5 nm are equivalent to 6.2 and 5.22 eV respectively indicating the ionic environment due to charge transfer transition in the complex caused by Fe^{3+} and Cl^- . The later assignment at 285 nm equivalent to 4.35 eV energy is not for a ionic environment but due to the sharing of electrons i.e. covalent bonding in the complex which is for the $p_\pi - d_\pi$ transition of Se_4N_3 ring in the complex. This opinion is also supported by the values of oscillator strength 'f' of the order of 10^{-5} (Table-1, Column-3) for the spin allowed laporte forbidden transition¹⁰. The low value of band gape energy, $\Delta E_g = 0.4351 - 0.4895$ eV and high values of number of conducting electrons, $N_c = 7.7287 - 8.8133 \times 10^5$ (Table-1, Column- 4&5) suggest the good conductive nature of complex.

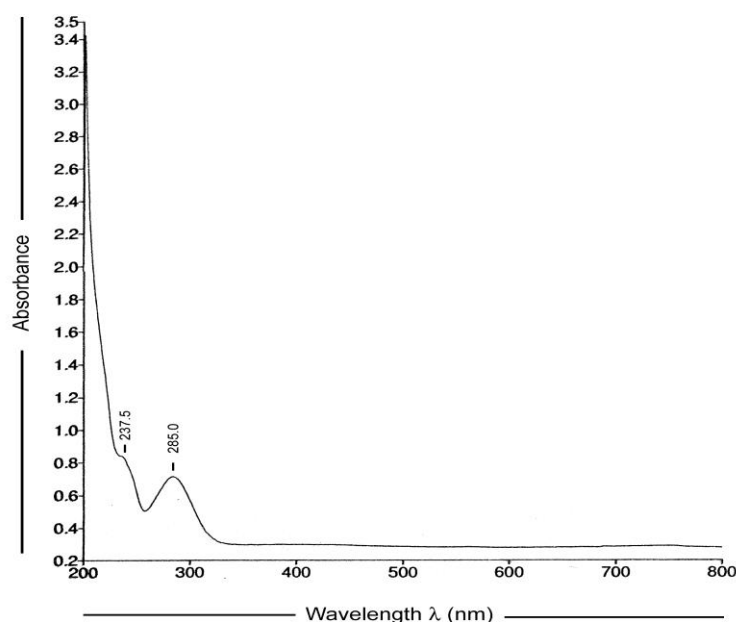


Fig. 1: U.V. spectrum of the Complex

E.P.R.spectra:

A broad peak of high intensity appeared at 2694.75 G in its E.P.R. spectrum explain the paramagnetism of complex. The paramagnetic character is supported by the

low value of $\mu_{eff} = 1.6492$ BM and magnetic susceptibility, $\chi_A = 1.1336 \times 10^{-3}$ e.s.u. (Table-1, Column- 9&10). The value of $g_x = g_y = 1.514$ (Table-1, Column-7) much less than two for a free electron indicate the availability of vacant '3d' energy shells to accept the lone pairs of electrons from N atom of Se_4N_3 ring forming coordinate linkage with Fe^{3+} . The higher value of $g_z = 2.5091$ (Table-1, Column-8) than $g = 2.003$ (for free electron). Infers the covalent linkage in the complex, showing the presence of Se_4N_3 ring. From the value μ_{eff} , the value of unpaired electron present found one which is for a $3d^5$ configuration of Fe(III).

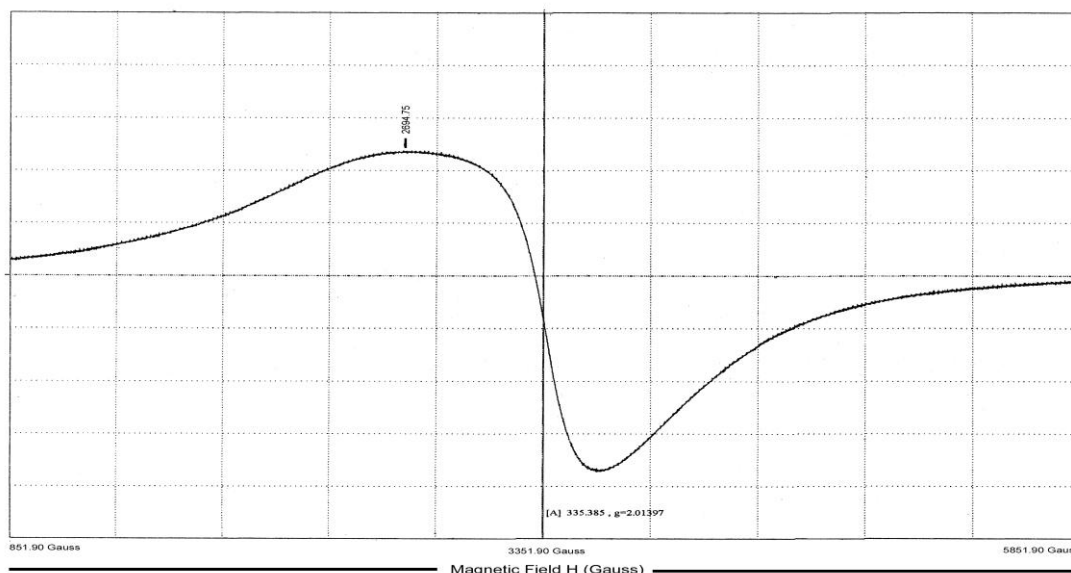


Fig. 2: E.P.R. spectrum of the Complex

Table-1: U.V. and E.P.R. Spectral Data of the Complex

U.V. Spectral Data					E.P.R. Spectral Data				
Band Assigned nm (cm ⁻¹)	Transition	f × 10 ⁵	ΔEg (ev)	N _c × 10 ⁵	Magnetic Field H (Gauss)	g _x = g _y	g _z	μ _{eff} (BM)	χ _A × 10 ⁻³ (e.s.u.)
1	2	3	4	5	6	7	8	9	10
200 (50,000)	C.T.	11.02613	-	-	2494.75	1.5140	2.5091	1.6492	1.1336
237.5 (42105.26)	C.T.	2.96626	0.4895	7.7287					
285 (35087.72)	p _π - d _π	2.56412	0.4351	8.8133					

X-ray Spectra:

From the X.R.D. spectrum of complex, recorded, in 2θ range 0° - 80° (fig-3) the value of $\sin^2 \theta$, miller indices, hkl, and inter planar distances 'd' are calculated (Table-2). The found value of 'd' are inclose agreement to that of theoretical ones. From these X.R.D. data, the values of axial ratio and axial angles, calculated, as $a_0 = 8.3848\text{\AA}$, $b_0 = 10.2692\text{\AA}$, $c_0 = 10.8247\text{\AA}$ and $\alpha = 120^\circ$, $\beta = 135^\circ$, $\gamma = 90^\circ$ are corresponding $a_0 \neq b_0 \neq c_0$ and $\alpha \neq \beta \neq \gamma \neq 90^\circ$ for the triclinic geometry of the complex.

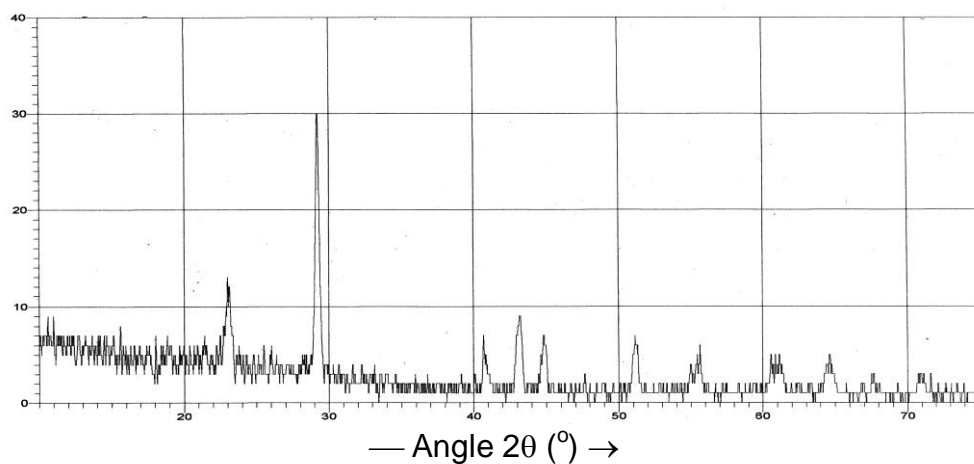


Fig. 3: X.R.D. Pattern of the Complex

Table-2: X-ray Diffraction pattern of complex

S. No.	$2\theta(^{\circ})$	$\sin^2 \theta$	$(h^2 + k^2 + l^2)Q$	hkl	d(\AA) Obs (theo)	$d_{hkl}(\text{\AA})$
	1	2	3	4	5	6
1	10.60	0.00853	$1 \times (0.00853)$	100	8.3134 (8.3387)	8.3849
2	15.60	0.01841	$2 \times (0.009205)$	110	5.6805 (5.6755)	5.9290
3	18.05	0.02460	$3 \times (0.00820)$	111	4.9146 (4.9076)	4.8410
4	23.01	0.03978	$5 \times (0.007956)$	210	3.8651 (3.8601)	3.7498
5	29.21	0.06358	$8 \times (0.007947)$	220	3.0573 (3.0537)	2.9645
6	33.16	0.08142	$10 \times (0.008142)$	310	2.7016 (2.6993)	2.6515
7	40.69	0.12087	$14 \times (0.008634)$	321	2.2173 (2.2149)	2.2409
8	44.82	0.14533	$17 \times (0.008548)$	322	2.0221 (2.0240)	2.0336
9	51.12	0.18615	$22 \times (0.008461)$	332	1.7867 (1.7533)	1.7876
10	55.64	0.21780	$25 \times (0.008712)$	430	1.6518 (1.6504)	1.6769
11	60.87	0.25660	$30 \times (0.008553)$	521	1.5218 (1.5208)	1.5308
12	64.76	0.28679	$34 \times (0.008435)$	530	1.4395 (1.4383)	1.4379
13	71.63	0.34242	$40 \times (0.008560)$	620	1.3174 (1.3168)	1.3257

$$Q_{av} = 0.0084525$$

$$a_0 = 8.3848\text{\AA}, b_0 = 10.2692\text{\AA}, c_0 = 10.8247\text{\AA} \text{ and } \alpha = 120^\circ, \beta = 135^\circ, \gamma = 90^\circ$$

.Antibacterial:

The complex inhibited against *S. aureus* (gram +ve) 16mm and *E. coli* (gram –ve) 9mm (fig-5) explain the effectiveness of the complex against these bacteria, which causes different disease such as diarrhea ,damage of tissue etc.

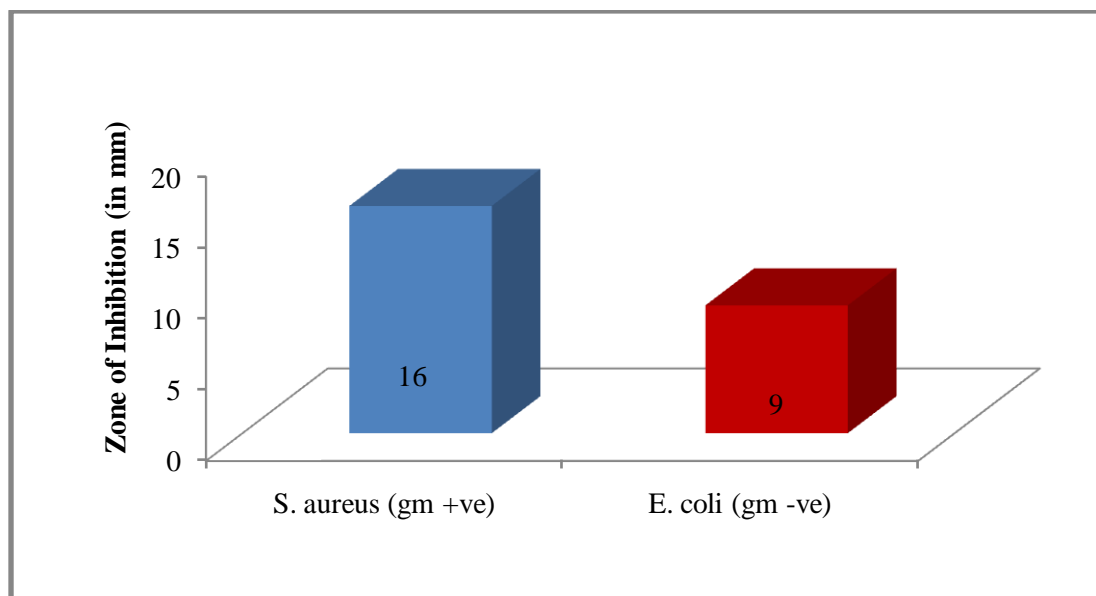


Fig. 5: Bactericidal effect of Complex

CONCLUSION

Thus from U.V., E.P.R. and X.R.D. studies, it is proved that complex possesses good conductive nature, and paramagnetic character having hexa coordinated array with triclinic geometry and its structure is confirmed (fig-4) as reported (loc.cit).

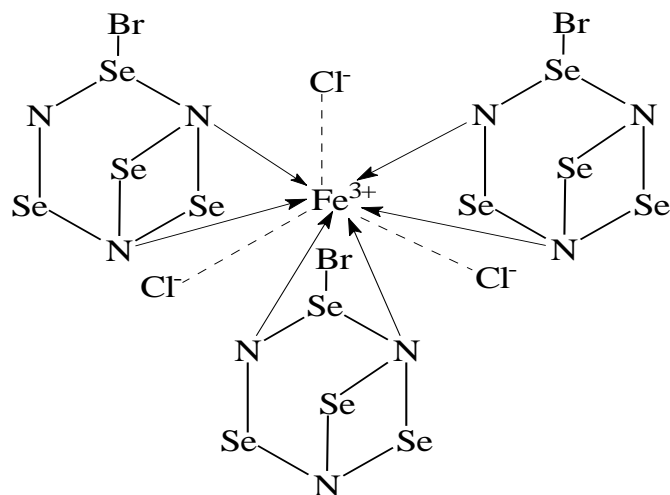


Fig. 4: Structure of Complex

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