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Synthesis and Characterization of Schiff Base Complexes of Eu (III) Chelates of Schiff bases Derived from Sulphanilamide and Furfuraldehyde

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Abstract: *The Schiff base complexes of Eu (III) derived from Sulphanilamide and aromatic aldehydes like N, N-dimethyl amino benzaldehyde and furfuraldehyde were synthesized and characterized by elemental analysis, conductivity measurements, infrared spectral measurements and antibacterial studies. The conductance measurements indicate that all the complexes are non-electrolytes. The IR results indicate the bidentate behaviour of the ligand coordinating through amino- nitrogen atom and azo methine- nitrogen to Eu^{3+} ion. All these complexes have moderate antibacterial activity.*

Keywords: *Antibacterial study, Conductivity measurements, Elemental analysis, IR spectra, Schiff base*

1. INTRODUCTION

Schiff bases are known to have many applications in organic and inorganic fields. Also, they were reported to have antibacterial, antifungal, and antitumor and antiherbicidal activities. Heterocyclic compounds such as pyridine, amino pyridine and related molecules are good ligands due to the presence of at least one ring nitrogen atom with a localized pair of electrons. The successful application has led to the formation of series of novel compounds with a wide range of physical, chemical and biological properties [1], spanning a broad spectrum of reactivity and stability. Hence it will be worthwhile to synthesize and characterize some new metal ligand complexes of Eu (III) with two different ligands based on sulphanilamide.

4[N-(4'- dimethyl amino benzalidene) amino] benzene sulphonamide (DBAB)

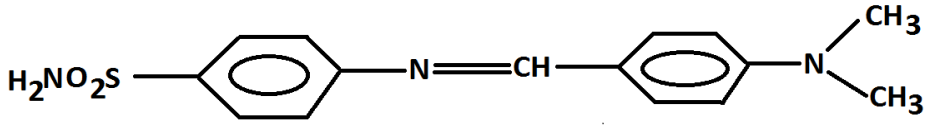

Fig.1 Structure of DBAB

4[N-(4'- furfuralidene) amino] benzene sulphonamide (FAB)

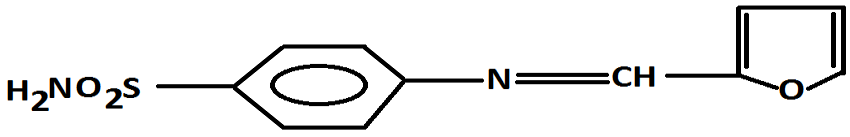

Fig.2 Structure of FAB

2. MATERIALS AND METHODS

2.1 Chemicals and Methods

All chemicals employed in the present study were of analytical grade. Solvents employed were either of 99% purity or purified by known laboratory procedures. Oxalate- oxide method is employed for the estimation of metal content in the complexes [2]. The nitrate content of the complexes was determined gravimetrically by using nitron reagent [3] and perchlorate content by Kurz's method [4,5]. Systronics-305 conductivity meter is used for conductivity measurements. Rast method was employed to determine the molar mass of complexes using biphenyl as solvent [6]. The antimicrobial studies of the complexes at varying concentration were done using two different bacteria viz. Staphylococcus aureus and Pseudomonas aeruginosa.

2.2 Synthesis of ligand

The ligands were prepared by standard methods [7]. Equimolar solutions of sulphanilamide and N, N-dimethyl amino benzaldehyde and furfuraldehyde in methanol were prepared and mixed together and refluxed for two hours. The resulting solutions were concentrated and cooled to get the solid crystals.

2. 3 Preparation of Schiff base metal complexes

The complexes were prepared by refluxing the acetonic solutions of the metal salts and the ligands in the mole ratio 1:3. The resulting mixture in each case was concentrated and cooled to get the solid complexes. They were washed repeatedly with benzene and petroleum ether to remove the excess of ligand. The coloured complexes were dried in vacuo over phosphorus (V) oxide.

3. RESULTS AND DISCUSSION

3. 1 Elemental analysis

All these complexes were insoluble in ether and partially soluble in nitrobenzene and moderately soluble in acetone and methanol. The results of analytical data of the complexes are shown in Table.1

Table.1 Analytical data of Eu (III) Complexes

Complex	Colour	Mol. Wt. Calcd (Found)	Percentage Analysis Calcd (found)				
			Metal	Anion	C	H	N
$[Eu(DBAB)_3(NO_3)_3]$	Black	1247 (1245)	12.2 (12.1)	14.9 (14.0)	43.3 (42.9)	4.1 (3.9)	13.5 (13.3)
$[Eu(FAB)_3(NO_3)_3]$	Black	1089 (1085)	13.9 (12.9)	17.1 (16.9)	36.4 (36.3)	3.0 (2.9)	11.6 (10.9)
$[Eu(DBAB)_3(ClO_4)_2]\ ClO_4$	Reddish Brown	1359 (1357)	11.2 (11.1)	22.0 (21.5)	39.7 (39.0)	3.8 (3.7)	9.3 (9.1)
$[Eu(FAB)_3(ClO_4)_2]\ ClO_4$	Brown	1201 (1199)	12.6 (11.9)	24.8 (23.9)	32.9 (31.9)	2.7 (2.1)	6.9 (5.9)

3.2 Electrical Conductivity

The observed molar conductance data indicate non-electrolytic nature of nitrato complexes in nitrobenzene because their conductivity values were in the range 5-10 $ohm^{-1}\ cm^2 mol^{-1}$ (Table 2), thus the nitrato complexes are non-electrolytes [8] and both primary and secondary ligands are in coordination sphere.

Table 2. Molar Conductance data of nitrato complexes

Complex	**Molar conductance in Nitrobenzene ($Ohm^{-1}\ cm^2\ mol^{-1}$)**
$[Eu(DBAB)_3(NO_3)_3]$	6.2
$[Eu(FAB)_3(NO_3)_3]$	7.5

Electrical conductance data of perchlorato complexes in nitrobenzene (shown in Table.3) corresponds to those of 1:1 electrolyte. It states that only two of the perchlorate ions were coordinated to the metal ion.

Table 3. Electrical Conductance data of perchlorato complexes

Complex	**Molar conductance in Nitrobenzene ($Ohm^{-1} cm^2 mol^{-1}$)**
$[Eu(DBAB)_3(ClO_4)_2] ClO_4$	25.4
$[Eu(FAB)_3(ClO_4)_2] ClO_4$	25.8

Based on these investigations, the present complexes can be formulated as
$[Eu\ L_3\ (NO_3)]$ and $[Eu\ L_3\ (ClO_4)_2]\ ClO_4$
Where, Ligand(L) = DBAB or FAB

3.3 IR Spectra

A strong band around 1630- 1660 cm^{-1} in IR by the two ligands indicates a stretching vibration of C = N. A downward shift of 20-25 cm^{-1} by this band proved the coordination of azo methine nitrogen to the metal ion. The sharp band around 3250-3290 cm^{-1} observed in all the ligands corresponds to the stretching vibration of NH_2 group. A downward shift of around 20 $cm^{-1}$of this band in spectra of complexes indicates the coordination of amino nitrogen to the metal ion.

A strong band observed around 1510 cm^{-1} in the ligand FAB is the characteristic of furan ring. A medium intensity band observed at 750 and 610 cm^{-1} corresponding to ν_{C-H} out of plane bending of furan ring. These bands were retained in all the complexes in the same position. This supports the non-participation of furan ring in coordination. The inability of coordination of oxygen atom of furan ring may be attributed to the utilization of the lone pair electron of oxygen atom for the aromatic sextet. Thus, all the ligands are acting as neutral bidentate coordinating through azomethine and amino nitrogen atoms.

The three bands of nitrato complexes observed at 1464,1384 and 1302 cm^{-1} due to ν_4, ν_1 and ν_2 modes of coordinated nitrate ion which are absent in the spectra of the ligands and other anionic complexes. The difference between ν_4 and ν_1 is 80 cm^{-1} which supports the unidentate coordination of nitrate ion [9].

The structure and bonding of metal complexes of weakly coordinating perchlorate ion have been reviewed by Rosenthal [10]. The perchlorato complexes show a strong band around 1177 cm^{-1} which is not present in the spectra of ligand and in other anionic complexes. This band is due to ν_4 vibrations of monodentate perchlorate ion. The bands at 1023,624 and 525 cm^{-1} can be assigned to ν_1, ν_3 and ν_5 vibrations of monodentately coordinated perchlorate ion. The ν_2 band is observed at 938 cm^{-1} as a medium intensity band. All these observations confirm the unidentate coordination of perchlorate ions.

3.4 Thermal Study

All the complexes undergo two stage decomposition around 250- 320^0C and 580-600^0C. The thermo gravimetric results indicate that all the nitrato complexes are quite stable upto 200^0C which shows the absence of water and other coordinated solvent molecules. The first stage decomposition is due to the dissociation of organic moiety and the second stage is the formation of metal oxide, Eu_2O_3 which is conformity with the percentage loss of mass obtained from TG curve and independent pyrolysis experiment. The initial decomposition temperature is frequently used to define the relative thermal stabilities of complexes. On the basis of experimental findings in the present course of study, the relative thermal stabilities of the chelates under examination can be given as, $[Eu(FAB)_3(NO_3)_3] > [Eu(DBAB)_3(NO_3)_3]$. FAB complex is more stable compared to DBAB complex.

3.5. Antimicrobial Activity

According to Overtone' concept of cell permeability, the lipid membrane that surrounds the cell favours the passage as only lipid soluble materials due to which lipo solubility is an important factor that controls antimicrobial activity. On chelation, the polarity of the metal ion is reduced to a greater extent due to the overlap of the ligand orbital and partial sharing of the positive charge of the metal ion with donor groups. Further, it increases the delocalization of π – electron over the whole chelate ring and enhanced lipophilicity of the complex. This enhanced the lipophilicity in turn enhances the

penetration of the complexes into lipid membranes and blocking of metal binding sites on the enzymes of the micro- organisms [12-15]. The antimicrobial activity assay was carried out by using disc diffusion method [11], where the zones of inhibition are measured in mm. All the complexes were screened in various concentrations and their antibacterial activity against: Staphylococcus aureus and Pseudomonas aeruginosa was determined. The antibacterial activity was estimated based on the size of inhibition zone in the discs. Under identical conditions the complexes of Eu (III) with FAB are found to be more active compared to DBAB.

Table. 4 Antibacterial activity data of Eu (III) complexes.

Complex	**Zone of Inhibition (mm)**			
	Staphy. Aureus		Pseudo. Aeruginosa	
	5mg/ disc	10mg/ disc	5mg/ disc	10mg/ disc
$[Eu(DBAB)_3(NO_3)_3]$	6	7	6	7
$[Eu(FAB)_3(NO_3)_3]$	13	14	12	13
$[Eu(DBAB)_3(ClO_4)_2]\ ClO_4$	7	9	7	9
$[Eu(FAB)_3(ClO_4)_2]\ ClO_4$	18	21	17	18

4. CONCLUSION

Schiff base complexes of Eu (III) were synthesized and characterized using Sulphanilamide. Characterization of complexes were done based on analytical and Spectral data. The non- electrolytic behaviour of complexes is revealed by molar conductivity studies. From spectral and thermal studies, a coordination number of 9 is assigned to nitrato complexes and 8 to perchlorato complexes. From antibacterial study, it is clear that perchlorate complexes are more active compared to nitrate complexes.

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