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Synthesis and Pharmacological Studies of Complexes of Eu (III) Ion with Some Schiff Bases of Sulphanilamide

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Abstract

Complexes of Eu (III) ion with corresponding Schiff bases of sulphanilamide with para Anisaldehyde and Vanillin were synthesized. Synthesized complexes were characterized by analytical studies, Conductivity measurements and IR spectra. Non- electrolytic properties of complexes were revealed by Molar conductivity measurements. IR spectra reveals the coordination of azo methine nitrogen to the metal ion. Antibacterial screening by disc diffusion method indicates the moderate activity of complexes..

Keywords- MBAB, HMBAB, Thermal studies, Antibacterial study

1. Introduction

Schiff bases and their coordination compounds have gained importance for over several years because of their application as models in biological, biochemical, analytical and antimicrobial system ^[1]. In the present study schiff base complex of nitrate and perchlorate of Eu (III) ion with two different ligands MBAB and HMBAB have been synthesized. These complexes were characterized by analytical studies, conductivity measurements and IR spectra. Also compared antimicrobial properties of Eu (III) complexes.

4[N-(4'- methoxy benzalidene) amino] benzene sulphonamide (MBAB)

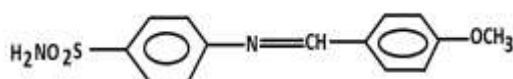


Fig.1 Structure of MBAB

4[N-(4'- hydroxyl-3'- methoxy benzalidene) amino] benzene sulphonamide (HMBAB)

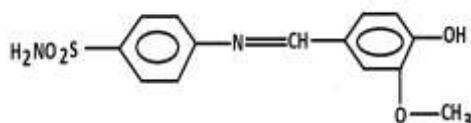


Fig.2 Structure of HMBAB

2. Materials and Methods

2.1 Chemicals and Methods

All the chemicals used were of GR/AR grade quality obtained from Merck chemicals. The metal content of the complexes was estimated by oxalate-oxide method ^[2]. The nitrate content of the complexes was determined gravimetrically by using nitron reagent ^[3] and perchlorate content by Kurtz method ^[4,5]. Conductance measurements were obtained using ystronics-305 conductivity meter. Rast method was employed to determine the molar mass of complexes using biphenyl as solvent ^[6]. The antimicrobial studies of the complexes and ligands 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 literature methods ^[7]. Equimolar solutions of sulphanilamide and para Anisaldehyde or vanillin in methanol were prepared and mixed together and refluxed for two hours. The resulting solution was 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

All these complexes were insoluble in ether and partially soluble in nitrobenzene and moderately soluble in acetone and methanol.

3.1 Elemental analysis

The formulae of complexes computed from analytical data is given in Table 1.

Table.1 Analytical data of Eu (III) Complexes

Complex	Colour	Mol. Wt. Calc'd (Found)	Percentage Analysis Calcd (found)				
			Metal	Anion	C	H	N
[Eu(MBAB) ₃ (NO ₃) ₃]	Reddish brown	1209 (1208)	12.6 (12.5)	15.4 (14.9)	41.7 (40.8)	3.5 (3.1)	10.4 (10.1)
[Eu(HMBAB) ₃ (NO ₃) ₃]	Brown	1257 (1255)	12.1 (11.9)	14.8 (14.6)	40.1 (39.9)	3.6 (3.4)	10.0 (9.9)
[Eu(MBAB) ₃ (ClO ₄) ₂]	Brown	1321 (1320)	11.5 (10.9)	22.6 (21.9)	38.1 (37.7)	3.2 (3.0)	6.4 (6.1)
[Eu(HMBAB) ₃ (ClO ₄) ₂]	Brown	1369 (1368)	11.1 (10.9)	21.8 (21.4)	36.8 (35.5)	3.3 (3.1)	6.1 (6.0)

3.2 Electrical Conductivity

The observed molar conductance data indicate non-electrolytic behaviour of nitrate complexes in nitrobenzene because their conductivity values were in the range 2-10 ohm⁻¹ cm²mol⁻¹ (Table 2), thus the nitrate complexes are non-electrolytes^[8] and both primary and secondary ligands are coordinated to the metal ion in nitrate complexes.

Table 2. Molar Conductance data of nitrate complexes

Complex	Molar conductance in Nitrobenzene (Ohm ⁻¹ cm ² mol ⁻¹)
[Eu(MBAB) ₃ (NO ₃) ₃]	6.58
[Eu(HMBAB) ₃ (NO ₃) ₃]	10.1

Electrical conductance data of perchlorato complexes in nitrobenzene (shown in Table.3) corresponds to those of 1:1 electrolyte suggesting

that primary ligand and only two of the perchlorate ions were coordinated to the metal ion in perchlorato complexes.

Table 3. Molar Conductance data of perchlorato complexes

Complex	Molar conductance in Nitrobenzene (Ohm ⁻¹ cm ² mol ⁻¹)
[Eu(MBAB) ₃ (ClO ₄) ₂]	27.2
[Eu(HMBAB) ₃ (ClO ₄) ₂]	26.3

Based on these investigations, the present complexes can be formulated as [Eu L₃(NO₃)₃] and [Eu L₃(ClO₄)₂] ClO₄ Where, Ligand(L) = MBAB or HMBAB

3.3 IR Spectra

IR spectrum of the two ligands exhibit a strong band around 1630- 1660 cm⁻¹. These are assigned to stretching vibration of C = N. This band undergoes a downward shift by 20-25 cm⁻¹ in the spectra of complexes. This clearly indicates 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 NH₂ group. This band undergoes a downward shift around 20 cm⁻¹ in spectra of complexes. This proves the coordination of amino nitrogen to the metal ion. The band at 1240 cm⁻¹ in the spectra of ligands, MBAB and HMBAB is for νOCH₃ which is retained in the spectra of complexes without any change of position. This shows that OCH₃ group of the ligand is not involved in bonding. A broad band observed in the region 3100-3300 cm⁻¹ for the ligand HMBAB and its complexes is assigned to νOH vibration.

The nitrate complexes observed three bands at 1464,1384 and 1302 cm⁻¹ due to ν₄, ν₁ and ν₂ modes of coordinated nitrate ion which are absent in the spectra of the ligands and other anionic complexes. The difference between ν₄ and ν₁ 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 $^{\circ}\text{C}$ and 580-600 $^{\circ}\text{C}$. The thermo gravimetric results indicate that all the nitrate complexes are quite stable upto 200 $^{\circ}\text{C}$ 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, $[\text{Eu}(\text{MBAB})_3(\text{NO}_3)_3] > [\text{Eu}(\text{HMBAB})_3(\text{NO}_3)_3]$. MBAB complex is more stable compared to HMBAB 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. Results showed that perchlorate complexes

exhibited significant antibacterial profile and more action (Shown in Table.4). Others show moderate activity.

Table. 5 Antibacterial activity data of Eu (III) complexes.

Complex	Zone of Inhibition (mm)			
	Staphy. Aureus		Pseudo. Aeruginosa	
	5mg/ disc	10mg/ disc	5mg/ disc	10mg/ disc
$[\text{Eu}(\text{MBAB})_3(\text{NO}_3)_3]$	8	12	9	10
$[\text{Eu}(\text{HMBAB})_3(\text{NO}_3)_3]$	-	-	6	8
$[\text{Eu}(\text{MBAB})_3(\text{ClO}_4)_2] \text{ClO}_4$	11	18	12	14
$[\text{Eu}(\text{HMBAB})_3(\text{ClO}_4)_2] \text{ClO}_4$	6	6	7	11

4. Conclusion

Complexes of Eu (III) with schiff bases derived from Sulphanilamide were synthesized. Characterization of the complexes were done based on analytical and Spectral studies. The non-electrolytic behaviour of complexes are revealed by electrical conductivity studies. The coordination behaviour of both primary and secondary ligand were ascertained by spectral and thermal studies. From antibacterial study, it is clear that perchlorate complexes are more active compared to nitrate complexes of MBAB and HMBAB

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