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Synthesis and Characterization Studies of Schiff base Complexes of Chromium (III)

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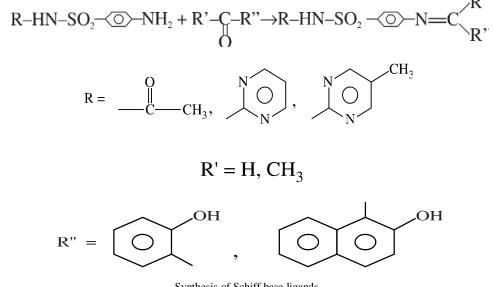
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ABSTRACT

M(III) complexes of chromium with Schiff base, derived from sulpha drugs and aldehyde / Ketone. These Complexes were characterized with the help of elemental analyses, conductance measurements, magnetic susceptibility measurements and IR spectral studies. The biological activities of these complexes have been tested invitro to evaluate their activity against gram (+) and gram (-) bacteria. Complexes for Escherichia coli (-), Staphylococcus aureus (+), Pseudomonas capacicola (-) were found, to be more active than ligands.

Graphical abstract



Synthesis of Schiff base ligands

Keywords: Cr (III) Complexes, NOH donor, antibacterial activity.

INTRODUCTION

One of the most characteristics features of the chemistry of chromium is the great ability of Cr (III) to form coordination compounds [1, 2]. Chromium (III) complexes of mixed-ligands have multifaceted importance [3, 4]. Tremendous biological significance of chromium (III) complexes has also been reported. Schiff bases [5, 6]are considered as a very important class of organic chemistry[7] which have wide applications in many biological aspects. Some Schiff bases were reported to possess antibacterial, antifungal and antitumor activities [8]. Due to their multiple implications the transition metal complexes [9] with Schiff bases as ligand are of paramount scientific interest. Schiff bases with donors (N, O) have structure similarities with natural biological system [10] and due to the presence of imine group (-N=CH-) are utilized in elucidating the mechanism of transformation and racemization reaction in biological systems [11].

MATERIALS AND METHODS

Synthesis of Schiff base ligand: Schiff base derived from sulpha drugs and aldehydes / ketones. To obtain these ligands, 2- hydroxy-1- napthaldehyde, o-hydroxy acetophenone and salicylaldehyde were mixed with sulpha drugs viz. sulphamereazine, sulphadiazine and sulphacetamide in 1:1 molar ratio in the medium of ethanol and refluxed for five hours. On cooling, crystals separated out which were further purified by recrystallization from acetone figure-1.

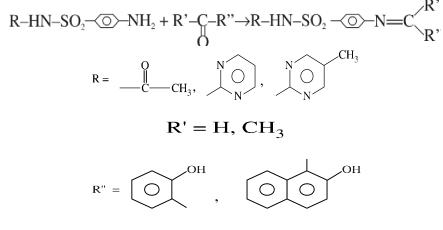


Figure 1. Synthesis of Schiff base ligands

Synthesis of Chromium (III) complexes: Chromium Compound and ligand were taken 1:2 molar ratio. Chromium (III) Chloride adduct [$CrCl_3$ (thf) ₃] was dissolved in dry methanol the mixture was heated under reflux for 2 h and the solution was concentrated by removing the solvent on a vacuum pump. It was then left overnight in a refrigerator. The product so obtained was filtered and washed repeatedly finally dried in vacuo at 40- 60°C. The details of the analyses and physical characteristic of the resulting products are given table 1.

$$[CrCl_3(thf)_3] + 2NO \xrightarrow{MeOH} [CrCl_2(NO)_2]Cl + 3THF$$

RESULTS AND DISCUSSION

All the complexes are colored solids, soluble in DMF and dimethyl sulphoxide (DMSO) but insoluble in benzene, chloroform and carbon tetrachloride.

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					Elemental Analysis (%)			
Chromium Compound	Ligand	Molar Ratio	Products Color and States	M.P (°C)	N Found (Cacld.)	S Found (Cacld.)	Cl Found (Cacld.)	Cr Found (Cacld.)
CrCl ₃ (thf) ₃	$C_{18}H_{16}N_4O_3S$ (L ¹ H)	1:2	$\begin{array}{c} CrCl_3\\ (C_{18}H_{16}N_4O_3S)_2\\ Green Solid \end{array}$	186	12.44 (12.52)	7.10 (7.17)	11.80 (11.89)	5.74 (5.81)
CrCl ₃ (thf) ₃	$C_{13}H_{12}N_2O_3S$ (L ² H)	1:2	$\begin{array}{c} CrCl_3\\ (C_{13}H_{12}N_2O_3S)_2\\ Green Solid \end{array}$	178	7.80 (7.88)	8.96 (9.02)	14.90 (14.96)	7.24 (7.31)
CrCl ₃ (thf) ₃	C ₁₆ H ₁₈ N ₄ O ₃ S (L ³ H)	1:2	$\begin{array}{c} CrCl_3\\ (C_{16}H_{18}N_4O_3S)_2\\ Blackish \ Green\\ Solid \end{array}$	152	13.10 (13.17)	7.48 (7.54)	12.42 (12.50)	6.04 (6.11)
CrCl ₃ (thf) ₃	C ₁₉ H ₁₈ N ₄ O ₃ S (L ⁴ H)	1:2	$\begin{array}{c} CrCl_3\\ (C_{19}H_{18}N_4O_3S)_2\\ Dark \ Green \ Solid \end{array}$	133	12.08 (12.14)	6.90 (6.95)	11.46 (11.52)	5.58 (5.63)

Table 1. The details of the analyses and physical characteristics of the products.

Magnetic and Conductance measurements: Magnetic susceptibilities were determined by Gouy's method at the room temp. The observed values of magnetic moment of the complexes show that all these complexes are paramagnetic. Their effective magnetic moment has been found to be 3.62- 3.72 B.M. (Table 2).

The conductance measurement in DMF at the room temperature shows that the 1:1 complexes are almost non- electrolyte $(10^{-35} \text{ ohm}^{-1} \text{ cm}^2 \text{ mol}^{-1})$, whereas the molar conductance of 1:2 complexes falls in the range 76-90 ohm⁻¹ cm² mol⁻¹, indicating that these complexes behave as electrolytes. (Table2).

S.No	Chromium (III) Complexes	Magnetic Moments (B.M.)	Molar Conductance Ohm ⁻¹ cm ² mol ⁻¹
1.	$CrCl_3(L^1H)_2$	3.72	76.14
2.	$CrCl_3(L^2H)_2$	3.62	88.398
3.	$CrCl_3(L^3H)_2$	3.68	78.82
4.	$CrCl_3(L^4H)_2$	3.63	82.52

Table 2. Magnetic and Conductivity data of chromium (III) complexes

IR Spectra: A sharp and strong band in the region $1610 \pm 10 \text{ cm}^{-1}$ due to v (C= N) appears in the spectra of ligands. This band shifts to the higher frequency side owing to increase in >C=N bond order on coordination through nitrogen. The spectra of ligands display two broad bands in the region 3450-3200 cm⁻¹ attributes to v (OH) and v (NH). A strong band at 1250cm^{-1} in free ligands due to v (C-O) shifts to higher frequency side, this further confirms the chelation through oxygen atom. New band at 480-490 cm⁻¹,360-370 cm⁻¹ and 225-265 cm⁻¹ due to v(Cr \leftarrow O), v(Cr-Cl) and v(Cr \leftarrow N) appears in the spectra of complexes.

APPLICATIONS

Antibacterial activities: All the synthesized ligands and chromium complexes were screened invitro applying paper disc plate method technique for antibacterial activity against gram positive and gramnegative bacteria{*Escherichia coli* (-), *Pseudomonas capacicola*(-) and *Staphylococcusaureus* (+)} at the 500ppm and 1000ppmconcentrationStreptomycin was used for as a reference compound for antibacterial activities. The antibacterial studies suggested that the Schiff base are biologically active and their chromium complexes showed significantly enhanced antibacterial activity against microbial

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strains in comparison to the free ligands. It has been observed that the chromium complexes showed increased zone of inhibition of the bacterial strain (Table 3) as compared to ligands. The formation of the coordination compounds can be used as the prospective antibiotic agents against some known pathogenic organism and can be used as marketed drugs.

	Diameter of inhibition zone (mm)							
Compounds	E.coli (-)		Staphylococcus		Pseudomonas			
- · · · · · · · · · · · · · · · · · · ·			aureus (+)		capacicola (-)			
	500 ppm	1000 ppm	500 ppm	1000 ppm	500 ppm	1000 ppm		
$L^{1}H$	4	6	5	7	-	5		
$CrCl_3(L^1H)_2$	5	7	6	9	-	6		
L ² H	3	5	4	6	4	6		
$CrCl_2(L^2H).2H_2O$	4	6	5	8	5	7		
L ³ H	5	7	4	8	6	7		
$CrCl_3(L^3H)$	7	9	5	9	7	9		
L^4H	4	6	5	7	5	6		
$CrCl_3(L^4H)$	6	8	7	9	6	8		
Streptomycin	15	18	15	17	10	14		

Table 3. Antibacterial screening data of the ligands and chromium (III) complexes inhibition zone (mm) after 24 h(Conc. in ppm)

CONCLUSIONS

The newly synthesized Schiff base and Chromium (III) Schiff base complexes have been tested on the various pathogenic gram positive and gram-negative bacteria, such as *E. coli, Staphylococcus aureus* and *Pseudomonas capacicola*. The results show that all these complexes inhibit the growth of both gram positive as well as gram negative bacteria at different concentrations. The results of these investigation show that the complexation increase the activity as the above metal complexes are more active than ligands.

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