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Synthesis, Characterisation and Antibacterial Activities of Titanium(III) Complexes with Schiff Bases Derived from Benzofuran

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ABSTRACT

Titanium(III) chloride reacts with schiff bases derived from benzaldehydes/acetophenone with 3-amino-2-benzofuran carboxamide / ethyl-3-amino-2-benzofuran carboxylate to yield complexes having general composition $[Ti.2L.2H_2O]Cl_3$ (where L=schiff base). The ligands and their corresponding Ti(III) complexes have been characterised by elemental analyses, molar conductance, magnetic susceptibility, IR & electronic spectra and TGA. The ligands act in a bidentate manner. Based on these studies octahedral geometries have been proposed for these complexes. The ligands and their Ti(III) complexes have screened for antibacterial activities.

Key words: Benzofuran, Schiff base, Octahedral and Antimicrobial activity.

INTRODUCTION

The compounds with benzofuran moiety have aroused much interest because of their biological importance¹. These compounds have a number of applications in both quantitative as well as qualitative analysis^{2,3}. Several transition metals also play many important roles in biological processes⁴. Keeping these facts in view, a number of schiff bases containing benzofuran moiety have been prepared and characterized. Their complexation behaviour towards Ti(III) has also been investigated.

MATERIAL AND METHODS

All the chemicals and reagents used were of AR grade or equivalent purity. Titanium(III) chloride was prepared by standard method given in chemical literature.

Synthesis of Schiff bases

3-amino-2-benzofuran carboxamide and ethyl-3-amino 2-benzofuran carboxylate were synthesised by known procedure⁵. The Schiff bases were prepared by refluxing the mixture of ethanolic solutions of corresponding benzaldehydes/

acetophenone and 3-amino-2-benzofuran carboxamide/ ethyl-3-amino-2-benzofuran carboxylate in 1:1 molar ratio in the presence of few drops of glacialacetic acid. The resulting schiff base was separated by filtration and washed repeatedly with ethanol and recrystallised from suitable solvent. The purity of the sample was tested by TLC.

Preparation of Complexes

Titanium(III) chloride (0.01 mole) was refluxed with schiff base (0.02 mole) in ethanolic medium for 3-4h. The complex that separated was filtered, washed with ethanol and dried in vacuum.

Analysis and measurements

The micro analysis of the complexes was carried out at Microanalytical laboratory at CDRI, Lucknow. The metals and chloride were estimated by standard methods.

The conductivity measurements were made on ELICO CL-82 Conductivity Bridge with a dip type conductivity cell. The magnetic susceptibility measurements were carried out at room temperature using Guoy's balance. Electronic spectra were recorded in DMF using Beckmann spectrophotometer. IR spectra of ligands and their complexes in KBr phase were recorded in the region 4000-400 cm^{-1} on a Perkin Elmer Infrared spectrophotometer.

RESULTS AND DISCUSSION

The analytical data suggested 1:2 (M:L) stoichiometry for all the complexes. All the complexes are amorphous in nature and stable towards atmosphere with high melting points (>300°C). The molar conductivity values in DMSO and DMF (10^{-3} M) indicating 1:3 electrolytic behaviour (Table 2.) The complexes are soluble in

Table 1: Physical data of the ligands

S. No.	Molecular Formula of the Ligand	m.p. (°C)	Yield, % (solvent of crystallization)	%Found (Calculated)		
				C	H	N
1	C ₁₆ H ₁₂ N ₂ O ₂	212	88 (ethanol)	72.71 (72.73)	4.50 (4.54)	10.60 (10.61)
2	C ₂₁₆ H ₁₁ N ₂ O ₂ Cl	204	85 (benzene +pet.ether)	64.29 (64.30)	3.50 (3.68)	9.41 (9.43)
3	C ₁₇ H ₁₄ N ₂ O ₃	194	90 (ethanol)	69.37 (69.39)	4.80 (4.82)	9.53 (9.54)
4	C ₁₆ H ₁₁ N ₃ O ₄	230	92 (aq.DMF)	62.11 (62.13)	3.61 (3.65)	13.58 (13.60)
5	C ₁₇ H ₁₄ N ₂ O ₂	208	83 (ethanol)	73.31 (73.38)	5.00 (5.03)	10.00 (10.07)
6	C ₁₈ H ₁₅ NO ₃	157	82 (ethanol)	73.80 (73.82)	5.16 (5.12)	4.81 (4.78)
7	C ₁₈ H ₁₄ NO ₃ Cl	168	84 (aq.Ethanol)	66.01 (65.95)	4.26 (4.27)	4.29 (4.27)
8	C ₁₉ H ₁₇ NO ₄	158	78 (benzene)	70.63 (70.59)	5.29 (5.26)	4.36 (4.33)
9	C ₁₈ H ₁₄ N ₂ O ₅	167	81 (aq.DMF)	64.02 (63.95)	4.11 (4.14)	8.31 (8.28)
10	C ₁₉ H ₁₇ NO ₃	160	87 (aq.Ethanol)	74.20 (74.26)	5.50 (5.53)	4.51 (4.56)

Table 2: Characterisation of metal complexes (obs.values)

S. No. of the complexes	Molecular Formula	Colour	m.p. (°C)	Elemental Analyses (%)				Metal	Mag. Moment (B.M.)		Molar conductance	
				C	H	N	Cl		DMSO	DMF		
1	[Ti(C ₁₆ H ₁₂ N ₂ O ₂) ₂ .2H ₂ O]Cl ₃	Yellow	250	53.31	3.87	7.72 (53.48)	14.50 (3.89)	6.61 (7.79)	1.69 (14.76)	115 (6.68)	110	
2	[Ti(C ₁₆ H ₁₁ N ₂ O ₂) ₂ .2H ₂ O]Cl ₃	Dark Yellow	271	53.36 (53.41)	3.75 (3.79)	7.69 (7.71)	14.68 (14.72)	6.59 (6.61)	1.71	125	120	
3	[Ti(C ₁₇ H ₁₄ N ₂ O ₃) ₂ .2H ₂ O]Cl ₃	Yellow		51.79 278	3.88 (51.83)	7.19 (3.92)	13.82 (7.32)	6.17 (13.87)	1.70 (6.28)	120	125	
4	[Ti(C ₁₆ H ₁₁ N ₃ O ₄) ₂ .2H ₂ O]Cl ₃	Yellowish orange	280	47.18 (47.46)	3.29 (3.33)	15.78 (15.82)	5.85 (5.93)	13.00 (13.10)	1.74	130	115	
5	[Ti(C ₁₇ H ₁₄ N ₂ O ₂) ₂ .2H ₂ O]Cl ₃	Yellow	256	53.96 (54.09)	3.95 (4.09)	7.54 (7.65)	6.48 (6.55)	14.39 (14.48)	1.74	125	120	
6	[Ti(C ₁₈ H ₁₅ NO ₃) ₂ .2H ₂ O]Cl ₃	Yellow	262	54.16 (54.25)	4.49 (4.52)	3.69 (3.72)	6.29 (6.38)	13.98 (14.04)	1.71	135	140	
7	[Ti(C ₁₈ H ₁₄ NO ₃) ₂ .2H ₂ O]Cl ₃	Light Yellow	306	53.89 (54.00)	4.21 (4.26)	3.68 (3.73)	6.36 (6.40)	13.92 (14.13)	1.69	125	135	
8	[Ti(C ₁₉ H ₁₇ NO ₄) ₂ .2H ₂ O]Cl ₃	Yellow	315	52.10 (52.50)	4.68 (4.75)	3.46 (3.50)	5.89 (6.00)	13.10 (13.25)	1.70	125	130	
9	[Ti(C ₁₈ H ₁₄ N ₂ O ₅) ₂ .2H ₂ O]Cl ₃	Deep Yellow	318	48.16 (48.45)	3.74 (3.80)	6.58 (6.65)	5.62 (5.70)	12.46 (12.58)	1.72	120	135	
10	[Ti(C ₁₉ H ₁₇ NO ₃) ₂ .2H ₂ O]Cl ₃	Yellow	320	54.61 (54.68)	4.92 (4.94)	3.62 (3.64)	6.11 (6.25)	13.74 (13.80)	1.70	125	130	

The calculated. Values are given in brackets

Table 3: Results of antibacterial activity of Schiff bases and their Cu(II)complexes

Ligand/ complex	Zone of inhibition		Ligand/ Complex	Zone of inhibition	
	<i>S.aureus</i>	<i>E.coli</i>		<i>S.aureus</i>	<i>E.coli</i>
DMF(control)	-	-	-	-	-
Ligand 1 Ti	-	-	Ligand 6 Ti	-	-
Ligand 2 Ti	-	-	Ligand 7 Ti	14 mm	-
Ligand 3 Ti	7 mm	-	Ligand 8 Ti	10 m	-
Ligand 4 Ti	-	8 mm	Ligand 9 Ti	-	-
Ligand 5 Ti	13 mm	10 mm	Ligand10 Ti	8mm	-
	-	7 mm		13 mm	-

DMF and DMSO and are insoluble in common organic solvents. Hence, the efforts for determining the molecular weight of complexes were not successful. The magnetic moments of the complexes fall in the range 1.69-1.74 B.M.

Electronic Spectra

The electronic spectra of the complexes exhibit a single broad band in the range of 19230-19600 cm^{-1} derived from ${}^2T_{2g} \rightarrow {}^2E_g$ transition for octahedral geometry(7).

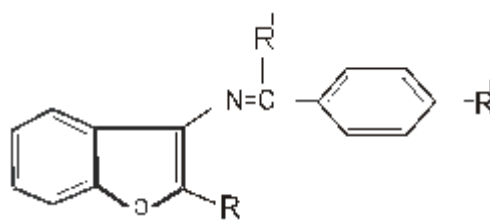
I.R. Spectra

The bands in the region 3460-3140 cm^{-1} are assigned to NH_2 vibrations of primary amide group in ligands practically remain unchanged in the spectra of the complexes suggesting nonparticipation of NH_2 group on chelation⁸. The schiff bases exhibit strong to medium intensity bands in the region 1590-1540 cm^{-1} (C=N stretching vibrations)⁹, undergo a negative shift of 20-40 cm^{-1} on complexation suggesting the involvement in coordination of azomethine group. The bands in the region 1700-1620 cm^{-1} (C=O stretch of amide or ester group) undergo a negative shift in the complexes by 80-45 cm^{-1} indicates the bonding through carbonyl group¹⁰. The bands in the region 1200-1175 cm^{-1} are attributed to C-O-C stretch of furan ring in free ligands¹¹. These bands remain unchanged indicating non-participation of furan ring oxygen on coordination. In the far IR

spectra of the complexes, the bands in the region 540-510 cm^{-1} and 485-400 cm^{-1} assigned to M-O stretch¹², M-N stretch^{13,14}. It can be concluded that all the ligands act as bidentate, with azomethine nitrogen and carbonyl oxygen atom as donor sites.

Anti-bacterial activity

Keeping in view of the potential biological activity, all the ligands and their copper complexes



Ligand	R	R'	R''
1	CONH ₂	H	H
2	CONH ₂	H	Cl
3	CONH ₂	H	OMe
4	CONH ₂	H	NO ₂
5	CONH ₂	Me	H
6	COOEt	H	H
7	COOEt	H	Cl
8	COOEt	H	OMe
9	COOEt	H	NO ₂
10	COOEt	Me	H

were tested against two bacteria namely staphylococcus aureus (S.aureus) and Escherichia coli(E.coli), which are representative type of gram positive and gram negative bacterial groups, respectively. The biological screening was conducted by filter paper disc method¹⁵. The Schiff base ligands and some of its metal complexes did not show any activity (Table 4). However, the complexes of ligands II, V and VI and VII showed promising results against S.aureus. Thus, these compounds may prove to be good antibacterials against gram positive bacteria

CONCLUSION

On the basis of studies performed octahedral geometry may be suggested for all these Ti(III) complexes.

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