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Some new Titanium (III) complexes of nitrogen and sulphur containing Bidentate ligands possessing good antibacterial activity.

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ABSTRACT-
Complexes of Titanium (III) salts with Bis (Cinnamaldehyde) semicarbazone, Bis (4-Chlorobenzaldehyde) Semicarbazone, Bis (3,4,5 Trimethoxybenzaldehyde) Semicarbazone, Bis (pyridine-2-aldehyde) Semicarbazone, Bis (Thiophene-2-aldehyde) semicarbazone have been prepared and characterized on the basis of infrared and electronic spectral measurement in conjunction with magnetic susceptibility measurements over a range down to liquid nitrogen temperature. Probable structures for these complexes in the solid state have been proposed. Various ligand field and nephelauxetic parameters (Dq,B,C& β) have been evaluated and found consistent with the six coordinated structure for TI (III) complexes.
KEY WORDS- *Semicarbazone, Schiff bases, Antibacterial activity, fungicidal activity.*

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I. INTRODUCTION-

A number of TI (III) complexes containing nitrogen bidentate ligands have been reported (1,2,3). Such complexesare mainly low spin and six coordinated. Many of the complexes of semicarbazones and other nitrogen donor ligands are widely employed in medicinal science (4). Since the discovery of the antitubercular activity of semicarbazones by Domagk (5) studies on their pharmacology have required a great dealof interest (6-10).

1. Experimental- All the chemicals used were of reagent grade. The chemicals used were, 4-cinnamaldehyde, 4-chloro benzaldehyde,3,4,5-trimethoxy benzaldehyde, Pyridine-2-aldehyde, Thiophene-2-aldehydeand the amine used was semicarbazide.
2. Preparation of Schiff Bases- The Schiff Bases were prepared by the Condensation of respective aldehyde with semicarbazide.
3. Preparation of Complexes- The complexes of TI (III) were synthesized by the addition of TI(III) chloride solution in T.H.F. to the ethanolic solution of Schiff base ligands. The precipitate thus obtained was repeatedly washed with T.H.F. It was filtered immediately and dried in vacuum dessicator over fused calcium chloride. The preparation of the complexes was carried out in an inert atmosphere of nitrogen in a glove bag.
4. Results and Discussion- All complexes are colored solid and stable. Elemental analysis of these complexes reveals that all the complexes have 1:2 metal ligand ratio. The molar conductance values at 10^{-3} Mdilutionof all the complexes of TI(III) indicate that all the complexes are 1:3 electrolytes.

Magnetic moment measurements were carried out at Gouy's balance. These values lie in the range of 1.39 to 1.82BM at room temperature (11-12). These values are in the range expected for spin only value for one unpaired electron.

5. Electronic spectra- The electronic spectra of all the complexes exhibits bands in the region 20,000 to25,000 cm^{-1} along with a shoulder on lower energy side. The appearance of the shoulder is due to the Jahn Teller distortion which affect 2eg excited state similarly as an Jahn Teller distortion of the four equatorial bands are short and the two axial bands are long. The degeneracy of 2eg level will be lifted in two components, $^2A_{1g}$ and $^2B_{1g}$ in which $^2A_{1g}$ will have lower energy than $^2B_{1g}$ are formed. The splitting of $^2T_{2g}$ ground state due to Jahn Teller effect will give rise the two components $^2B_{2g}$ and ^2Eg. The $^2B_{2g}$ component has lower energy than ^2Eg indicated by magnetic measurements. The splitting of $^2T_{2g}$ is of the order of 420-800 cm^{-1} as indicated by electronic spectroscopy. The energy separation between $^2B_{1g}$ and $^2B_{2g}$ levels is 10Dq which is the highest energy component. The electronic spectral assignments of the complexes of TI(III) with N-containing ligands specially Schiff bases have been given in table no.2
6. I.R. SPECTRA- The infrared spectra of the ligands show few important absorption bands at 1621 (vs) cm^{-1} may be assigned to C=N vibration (13). This absorption band shifts at 1630 (vs) cm^{-1} in the spectra of complex. This shift indicates that the coordination may have occurred through the nitrogen atoms of azomethine

group. Thus, Schiff base, the ligand seems to be acting in a bidentate manner. A new peak appears in the spectra of the complex at 745 cm^{-1}, this may be due to the coordination of two water molecules. The T.G.A. of the complex confirms the presence of two coordinated water molecules, as the percentage weight lost around 180°C corresponds to the loss of two water molecules (14-15).

7. Antibacterial activity- The antibacterial activity of TI(III) complexes was carried out against 24 hours cultures of four selected bacteria. The bacterial activity was performed by cup plate technique. The Agar cups were made by proceeded (12ml) melted Agar medium at 50°C by boring 1mm thick broth culture on a plate by 10mm cork borer. Two drops of the melted Agar were pipetted into it, incubated for 24 hours and zone of inhibition observed. Results obtained are presented in Table no. (3).

8. Fungicidal Activity- The fungicidal activities of complexes were evaluated by testing them against different species at different concentrations. The results of fungicidal screeningisrecorded in Table no. (4).

Table no.1

SI. No.	Name and molecular formula of the complex	color	M.P./ D.T. □	Elemental analysis					Molar Conductance $Ohm^{-1} cm^{-1} mole^{-1}$			Magnetic Moment in B.M.
				%ofC	% ofH	% of N	% of M	%ofCl	Methanol	DMF	DMSO	
1.	Bis(cinnamaldehyde) Semicarbazone TI(III) chloride $(C_{19}H_{17}N_3O)_2 2H_2O\ TiCl_3$	Yellow	305□	57.257 (56.012)	4.771 (4.512)	10.547 (9.246)	6.014 (5.961)	13.372 (12.262)	183.5	175.0	51.3	1.70
2.	Bis(4chlorobenzaldehyde) Semicarbazone Titanium (III) Chloride $(C_{15}H_{11}N_3OCl_2)_2 2H_2O\cdot TiCl_3$	Reddish Brown	285□	43.352 (42.905)	3.131 (2.269)	10.115 (9.084)	5.768 (4.875)	29.925 (28.865)	210.0	181.0	90.0	1.70
3.	Bis (3,4,5 trimethoxy Benzaldehyde) semicarbazone titanium (III) chloride $(C_{21}H_{25}N_3O)_2 2H_2O\ TiCl_3$	Dark Brown	270□	58.577 (57.711)	6.276 (5.505)	9.762 (8.32)	5.567 (4.994)	12.377 (11.163)	250.0	169.0	60.0	1.59
4.	Bis (Pyridine 2-aldehyde) semicarbazone TI(III) chloride $(C_{13}H_{11}N_5O)_2 2H_2O\ TiCl_3$	Yellowish Brown	265□	44.801 (43.461)	3.733 (2.905)	20.103 (19.044)	6.878 (5.610)	15.292 (14.192)	190.0	165.0	72.5	1.65
5.	Bis (Thiophene 2-aldehyde) semicarbazone TI(III) chloride $(C_{11}H_9N_3OS_2)_2 2H_2O\ TiCl_3$	Dark Yellow	355□	36.850 (35.695)	3.070 (2.945)	11.725 (10.534)	6.686 (5.405)	14.865 (13.298)	240.0	160.0	85.0	1.73

Table no.2

Electronic spectral bands of TI(III) Complexes of some Schiff bases (cm^{-1})

S.no	Name of compounds	$^2B_{1g} \leftarrow {}^2B_{2g}$	$^2A_{1g} \leftarrow {}^2A_{2g}$	C.T.	E	10Dq
1.	$(C_{19}H_{17}N_3O)_2 2H_2O\ Ticl_3$	17857	_	31250	_	17857
2.	$(C_{15}H_{11}N_3OCl_2)_2 2H_2O\ TiCl3$	21737	17391	25000	4398	21737
3.	$(C_{21}H_{25}N_3O)_2 2H_2O\ TiCl_3$	20833	16000	29412	_	20833
4.	$(C_{13}H_{11}N_5O)_2 2H_2O\ TiCl_3$	22727	17241	31250	5483	22727
5.	$(C_{11}H_9N_3OS_2)_2 2H_2O\ TiCl_3$	23256	18519	26680 30000	4737	23256

Table no.3

Antibacterial Screening in DMF

S.I. of compound	Diameter of zone of inhibition (mm)			
	S.Aureus	E.Coli	B. Subtilis	S. Pyrogens
1.	10	8.0	11.4	9.8
2.	9.2	7.0	10.1	9.6
3.	9.6	9.2	10.7	9.1

4.	9.5	9.4	10.6	9.4
5.	10	8.2	11.2	9.5
6.	9.7	8.2	9.8	9.2
7.	9.2	9.8	10.1	9.3
8.	10.1	9.4	11.3	9.4
9.	9.3	9.6	11.2	8.9
10.	9.6	8.8	10.8	9.0

Table no.4

Fungicidal Screening data average % inhibition of Spore germination after 72 hrs.

S.I. of compound	Alternaria Alternata (conc. In ppm)			Aspergillus niger (conc. In ppm)		
	100	500	1000	100	500	1000
1.	52	57	62	49	47	57
2.	49	52	60	41	50	56
3.	50	56	61	43	50	54
4.	51	53	62	46	49	52
5.	49	57	60	42	51	53
6.	51	57	62	40	57	58
7.	56	60	64	47	58	60
8.	48	54	67	49	54	59
9.	54	52	59	48	51	52
10.	53	55	60	43	52	53

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