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Studies of Transition Metal of Substituted Oxazole

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ABSTRACT:

The chemistry and wide range of pharmaceutical properties of oxazole, thiazole, oxazoline and thiazoline have attracted the attention of many workers. They are chiefly used as analgesic, nematocides bactericides and fungicide. Thiazole and its derivatives possess antimalarial, antifungal, antibacterial and antitubercular activities. Several heterocyclic compounds form complexes with transition metal ions and these complexes have shown importance in the field of medicine and analytical chemistry.

Key words: oxazole, thiazole, oxazoline and thiazoline

INTRODUCTION:

Recently a large number of pharmaceutical applications of oxazole and oxazole derivatives like hypertensive, analgesic, anti-inflammatory, antibacterial, antiviral, antitubercular, anticonvulsant, urinary trac infections sedative, cardiac stimulant, antihistaminic muscle relaxant and hypotensive, have been reported. Oxazoline derivatives have been of interest because of their plant growth regulating activity. It is well known that chloro compounds belonging to the azole class are used as fungicides. Several heterocyclic compounds are known which form complexes with metal ions. These metal complexes have shown importance in the field of medicine, analytical chemistry and biological activity. The activity of the drug increases when it is used in the form of metal complexes. Metal complexes play a significant role in the biological system. Survey of the literature revealed that several metal complexes are used as anticancer drugs, antiviral and antimicrobial agents and in the treatment of arthritic and inflammation. A number of metal complexes inhibit tumor growth. The inherent bio-potency, structural aspects and unique stereo and magnetic chemistry of transition metal and their complexes attracted the interest of several workers. Due to the wide range of applications of oxazole and many workers. Several workers have prepared and oxazoline have attracted attention of the characterised transition metal complexes formed by heterocylic ligands. Recently a few reports appeared in literature about the importance of metal complexes in the field of medicinal, analytical, industrial and biological process.

The survey of the literature revealed that no systematic work have been done so far on the structural and biological aspects of transition metal complexes of chloro oxazole, so it is worth while to prepare transition metal complexes of various chloro oxazole and study their structural and biological activity.

EXPERIMENTAL FINDING

(A) Preparation of the Ligand 2-Amino-4-phenyl-5-Chloro Oxazole : The ligand 2-Amino-4-phenyl-5chloro-oxazole was prepared according to the method reported in literature for oxazole.

2-Amino-4-		% fou	nd/Calc.				S 3 4 1
phenyl-5-phenyl chloro oxazole	С	Н	Ν	Cl	M.P.	°C	Solubility
$C_9H_7N_2OC_1$	55.43 (55.52)	3.61 (3.59)	14.50 (14.39)	18.30 (18.25)	190	aq.	alcoholic solution

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(B) Preparation and Isolation of 2-Amino-4-phenyl-5-chloro-oxazole complexes: Dichloro/Dinitrato/Diacetato and Sulphato BIS (2-Amino-4-phenyl-5-chloro-oxazole Mn (II) :

Ethanolic solution of hydrated Mn (II) salts were added to an ethanolic solution of ligand 2-Amino-4-phenyl-5chloro-oxazole (metal : ligand 1 : 2 ratio) and their pH was raised by adding few drops of dil. ammonia solution. The cream coloured precipitates immediately separated out but the complete precipitation obtained by refluxing the mixture for an hour on water bath. The product were filtered, washed with alcohol and dried in vaccuo. Chemical analysis corresponds to following molecular formulae.

Complex	% found / Calc.							
Complex	С	Н	Ν	Mn	Cl			
	41.92	2.74	10.90	10.68	27.54			
	(41.94)	(2.71)	(10.87)	(10.66)	(27.58)			
	38.01	2.48	14.76	9.69	12.52			
	(38.03)	(2.46)	(14.79)	(9.65)	(9.65) (12.50)			
	46.96	3.52	9.98	9.75	12.65			
	(46.98)	(3.56)	(9.96)	(9.77)	(12.63)			
	40.02	2.56	10.39	10.19	13.11			
	(40.00)	(2.59)	(10.37)	(10.17)	(13.14)			

Trichloro/Trinitrato/Triacetato and Trisulphato Tris (2-Amino-4-phenyl-5-chloro oxazole) Fe (III) :

Hot ethanolic solution of the ligand was mixed with hydrated Fe(III) salts in the same solvent and in 1 : 3 ratio of metal and ligand. The mixed solution was refluxed for two hours on a water bath. The reaction mixture was then concentrated to half of its volume and solid complexes were then filtered and washed kept for overnight. The crystals of complex seperate out with ethanol and finally with ether and dried over P_2O_5 . Elemental analysis led to the following molecular formulae.

Complex	% found / Calc.						
Complex	С	Н	Ν	Fe	Cl		
[Fe(C ₉ H ₇ N ₂ OCl) ₃] Cl ₃	43.22	2.84	11.22	7.40	28.59		
	(43.44)	(2.81)	(11.26)	(7.49)	(28.56)		
$E_{2}(C \parallel N \mid OC) \mid (NO \mid)$	39.20	2.45	15.24	6.70	12.85		
$[Fe(C_9H_7IN_2OCI)_3]$ (INO 3)3	(39.25)	(2.54)	(15.27)	(6.77)	(12.90)		
	48.42	2.52	10.20	6.81	13.02		
$[Fe(C_9H_7N_2OCI)_3]$ (CH ₃ COO) ₃	(48.50)	(2.57)	(10.29)	(6.84)	(13.05)		
$\mathbf{F}_{\mathbf{a}} (\mathbf{C} \mathbf{H} \mathbf{N} \mathbf{O} \mathbf{C}) 1 (\mathbf{S} \mathbf{O})$	41.32	2.65	10.78	7.14	13.62		
$[1^{\circ}c_{2}(C_{9}\Pi_{7}\Pi_{2}OCI)_{6}](SO_{4})_{3}$	(41.36)	(2.68)	(10.72)	(7.12)	(13.59)		

Cichloro/Dinitrato/Diacetato & Disulphato (2-Amino-4-phenyl-5-chlorophenyl) Oxazole Fe(II) : Complexes :

These complexes were synthesized following the above procedure except metal to ligand ration which were kept as 1 : 2 in each case. Elemental analyses correspond to the molecular formulae given below.

Complex		% found / Calculated							
Complex	С	Н	Ν	Fe	Cl				
$E_{2}(C \parallel N \cap C^{1}) \subset L^{1}$	41.80	2.68	10.84	10.88	27.49				
$[Fe(C_9H_7IN_2OCI)_2CI_2]$	(41.87)	(2.71)	(10.85)	(10.82)	(27.52)				
$[Fe(C_9H_7N_2OCl)_2(NO_3)_2]$	33.92	2.50	14.82	9.88	12.40				
	(37.97)	(2.46)	(14.76)	(9.81)	(12.48)				
$[\mathbf{F}_{\mathbf{e}}(\mathbf{C}, \mathbf{H}_{\mathbf{N}}, \mathbf{O}_{\mathbf{C}}]), (\mathbf{C}_{\mathbf{H}}, \mathbf{C}_{\mathbf{O}}, \mathbf{O}_{\mathbf{N}}),]$	46.88	3.58	9.92	9.98	12.58				
$[Fe(C_9 \Pi_7 \Pi_2 OCI)_2 (C\Pi_3 COO)_2]$	(46.90)	(3.55)	(9.94)	(9.92)	(12.61)				
$[\mathbf{E}_{\mathbf{A}}(\mathbf{C} \mid \mathbf{H} \mid \mathbf{N} \mid \mathbf{OC}]) \mid (\mathbf{SO} \mid)]$	39.89	2.50	10.31	10.38	13.15				
$[1^{e}(C_{9}\Pi_{7}\Pi_{2}OC_{1})_{2}(SO_{4})]$	(39.93)	(2.58)	(10.35)	(10.32)	(13.12)				

Dichloro/Dinitrato/Diacetato/And Disulphato (2 Amino-4-phenyl)-5-chloro oxazole) Co (II) Dihydrate Complexes :

These complexes were prepared by refluxing for one hour, the respective metal salts with ligand in 1 : 2 molar ratio in aqueous ethanolic medium, on concentrating, the complexes so formed were suctioned, filtered, washed with alcohol, ether and dried in vacuo.

Complex	% found / Calculated							
Complex	С	H	Ν	Со	Cl			
	38.86	3.22	10.01	10.67	25.61			
$[CO(C_9H_7IN_2OCI)_2CI_2]$.2H ₂ O	(38.92)	(3.24)	(10.09)	(10.62)	(25.59)			
	35.56	2.90	13.78	9.78	11.69			
$[CO(C_9\Pi_7IN_2OCI)_2(INO_3)_2].2\Pi_2O$	(35.52)	(2.96)	(13.82)	(9.70)	(11.67)			
	43.81	3.92	9.18	9.68	11.70			
$[CO(C_9\Pi_7\Pi_2OCI)_2(C\Pi_3COO)_2].2\Pi_2O$	(43.85)	(3.98)	(9.21)	(9.70)	(11.68)			
	39.75	3.36	10.22	10.80	13.09			
$[CO(C_{9}I_{7}I_{2}OCI)_{2}](SO_{4})].2\Pi_{2}O$	(39.71)	(3.30)	(10.29)	(10.83)	(13.05)			

Dichloro/Dinitrato/Diacetato and Disulphato Bis (2-Amino-4-phenyl)-5-chloro) oxazole Ni (II) Monohydrate :

The metal complexes were prepared by refluxing an aqueous solutions of appropriate Ni(II) salt with acetonic solutions of ligand in 1 : 2 ratio (metal : ligand) molar ratio for an hour on water bath. The reaction product were concentrated to half of its volumes and then kept for overnight. The complexes so formed were filtered, washed with water, acetone and finally with either and dried in vacuo.

Complex	% found / Calc.							
Complex	С	H	Ν	Ni	Cl			
	40.19	2.92	10.40	10.98	26.42			
$[1N1(C_9H_71N_2OC1)_2C1_2].H_2O$	(40.24)	(2.98)	(10.43)	(10.94)	(26.46)			
	36.64	2.78	14.22	9.90	12.08			
$[1N1(C_9H_71N_2OC1)_2(1NO_3)_2].H_2O$	(36.62)	(2.71)	(14.24)	(9.95)	(12.04)			
	45.28	3.72	9.51	10.08	12.18			
$[1N1(C_9\Pi_71N_2OC1)_2(C\Pi_3COO)_2.]\Pi_2O$	(45.22)	(3.76)	(9.59)	(10.05)	Cl 8 26.42 4) (26.46) 0 12.08 6) (12.04) 8 12.18 5) (12.16) 2 12.70 4) (12.64)			
	38.48	2.88	9.99	10.42	12.70			
$[101(C_9\Pi_7\Pi_2OCI)_2SO_4]\Pi_2O$	(38.45)	(2.84)	(9.96)	(10.44)	(12.64)			

Dichloro/Dinitrato/Diacetato/And sulphato Bis (2-Amino-4-phenyl)-5-chloro) oxazole Cu (II) :

These complexes were prepared following the above method. Elemental analyses correspond to the molecular formulae given below.

Compley		% found / Calculated						
Complex	С	H	Ν	Cu	Cl			
	41.29	2.69	10.78	12.20	27.18			
$[Cu(C_9\Pi_7IN_2OCI)_2]CI_2$	(41.25)	(2.67)	(10.70)	(12.14)	(27.13)			
	37.42	2.48	14.52	11.10	12.34			
$[Cu(C_{9}\Pi_{7}\Pi_{2}OCI)_{2}](\Pi O_{3})_{2}$	(37.46)	(2.42)	(14.57)	(11.02)	(12.31)			
$[Cu(C_9H_7N_2OCl)_2] (CH_3COO)_2$	46.22	3.42	9.90	11.17	12.50			
	(46.27)	(3.50)	(9.81)	(11.13)	(12.44)			
	39.39	2.54	10.24	11.52	12.90			
$[Cu (C_{9}I171N_{2}OCI)_{2}]SO_{4}$	(39.37)	(2.55)	(10.20)	(11.58)	(12.94)			

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RESULTS AND DISCUSSION:

From the table it is clear that stoichiometry composition, in all the cases metal-ligand ratio is 1:2 except Fe (III) complexes where 1:3.

INFRARED SPECTRAL STUDIES:

In general the absorption frequencies of ligand get affected after complex formation. Here also absorption band due to amino and $\Box \Box$ (CO) are effected most. The main absortion bands of the ligand are lying in the region 3450-3400, 3335-3300 and 1560-1550 cm⁻¹. These are due to antisymmetric and symmetric $\Box \Box \Box$ (NH) and N=C-O-ring stretching frequencies. The characteristics band of oxazole system 17,18,19 were observed at 1640-1610, 1600-1590 and 1540-1535 cm⁻¹. The absorption band due to \Box CH. \Box C=0.-C-O-C and \Box (CCl) were observed in the region 3060-3000, 1620-1590, 1150-1100, 800-600 cm^{-1} . The presence of these band support the aromatic character of the ligand. A weak absorption band observed in the ligand as well as in the complexes were observed in the region 1590-1665 assigned to phenyl absorption band. On comparison of the spectra of metal complexes with those of the ligand it is observed that on complexation the \Box (C=N) stretching frequencies observed at 1470-1450 cm⁻¹, in the free ligand remain practically unchanged supporting that the ring nitrogen does not take any part in complex formation or coordination. The antisymmetric and symmetric (NH) stretching frequencies appear in the region 3450-3400 and 3335-3300 cm⁻¹ in the free ligand get shifted to the lower frequencies after complex formation. This shows that the nitrogen of the amino group is taking part in coordination. Considerable lowering in these frequencies along with the changes in the deformation, wagging and rocking amino vibrations suggest that metal are coordinated through nitrogen of the amino group because, the lone pair of electrons on the nitrogen of the amino group are more basic than those on nitrogen in the ring system, and is used to explain both the reasons of high electron density and formation of coordinate linkage In the present complexes bands observed in the region 370-270 cm⁻¹ were assigned to M-O band. This shows that the oxygen of the oxazole ring is taking part in the coordination.

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