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Synthesis and Characterization of 2-(8-Quinololinol-5-yl) - amino methyl-3-(4- Methyl Phenyl)-5- (4-chloro Phenyl)-Pyrazoline and its Metal Complexes with Co(II), Ni(II) and Mn(II) Ions and Study of their Antimicrobial Activities

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ABSTRACT

Complexes of 2-(8-Quinololinol-5-yl) - amino methyl-3-(4- Methyl Phenyl)-5- (4-chloro Phenyl)-Pyrazoline with Co(II), Ni(II) and Mn(II) have been synthesized and characterized using elemental analysis, IR spectra, PMR spectra and antimicrobial activity. These studies revealed that they are having octahedral geometry of the type $[ML_2(H_2O)_2]$. The compounds show net enhancement in activity on coordination of metals with ligand but moderate activity as compared to standard drugs.

Key Words : pyrazoline, ligand, chalcones , chelates.

INTRODUCTION

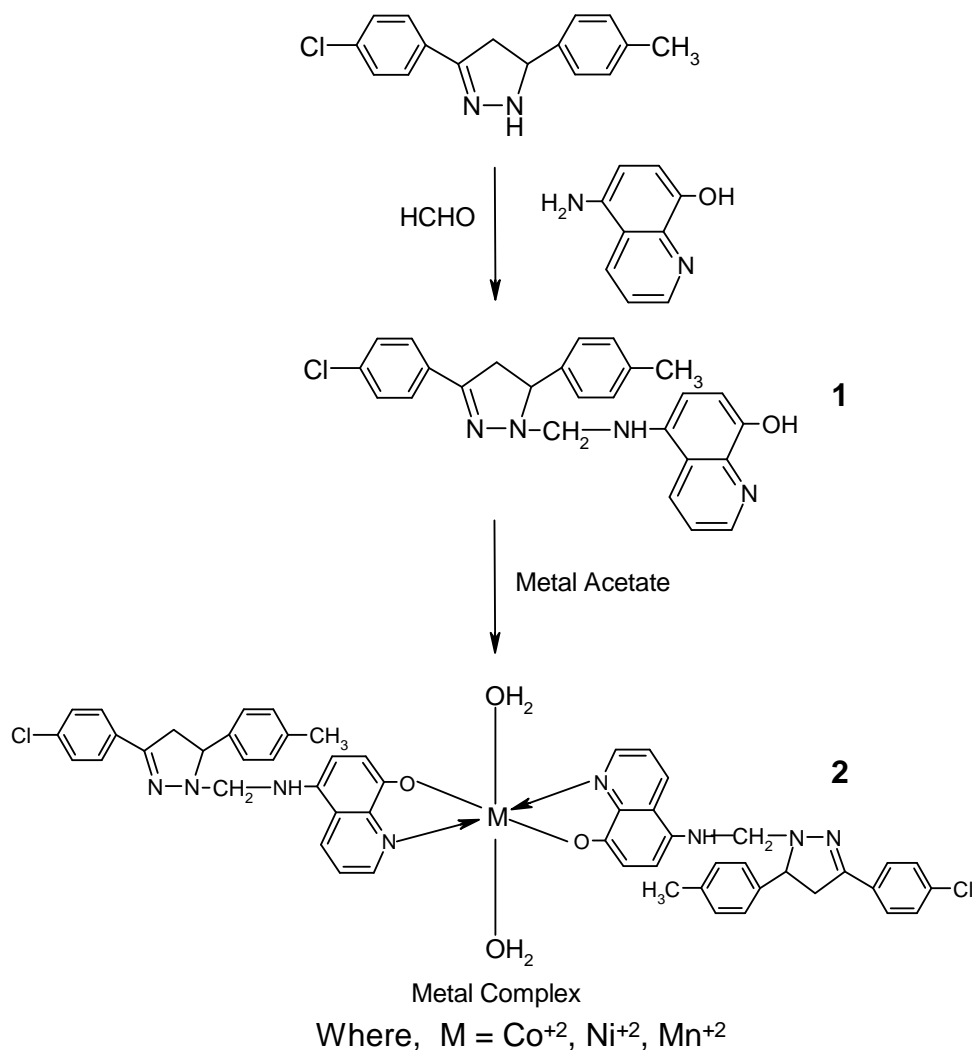
With a variety of biological activity, chalcones are useful in pharmaceuticals. They are associated with different biological activities like insecticidal[1], anticancer [2], anti-inflammatory[3], bactericidal[4], fungicidal[5], antiviral[6], antitumor[7] and antiulcer[8] Synthesis of pyrazolines has been also stimulated by the fact that some of their derivatives were found to possess important bioactivities. Especially their antimicrobial [9], immunosuppressive [10] and central nervous system activity [11] should be emphasized.

MATERIALS AND METHODS

Experimental

Melting points were taken in open capillary tube and were uncorrected. IR spectra (KBr) were recorded on Nicollet FTIR 760 and PMR spectra were recorded on Bruker NMR spectro-

photometer. PMR chemical shifts are recorded in δ value using TMS as an internal standard in $CDCl_3/D_6$ -DMSO. Purity of the compounds were checked by tlc on silica- G plates. The fungicidal activity of all the compounds was studied at 1000 ppm concentration in vitro. Plant pathogenic organisms used were *Penicillium expansum*, *Botrydepladia thiobromine*, *Nigrospora Sp.*, *Trichothesium Sp.*, and *Rhizopus nigricum*. Anti bacterial activities were tested by Agar Cup method.



Preparation of 2-(8-Quinolinol-5-yl) - amino methyl-3-(4- Methyl Phenyl)-5- (4-chloro Phenyl)-Pyrazoline. [HL] (1).

A mixture of 3-(4-methyl phenyl)-5-(4-chloro phenyl) -2H- Pyrazoline (0.01 mole) and formaldehyde (40%, 1.5 ml) in ethanol (20 ml) was stirred at room temp. With a solution of 5-Amino-8-Quinolinol (0.01 mole) in ethanol (10 ml) for 30 min. The solid product that separated out on standing for a 1 hrs was collected by filtration, washed with ethanol & dried. It was recrystallized from ethanol to yield the ligand compounds having m.p- 237°C. (Uncorrected). The yield of the product was 67 % .Found: C(69.9%) H(5.2%) N(12.5%) Cl(7.8%), Calcd. for $C_{26}H_{23}N_4OCl$: C(70.5%) H(5.2%) N(12.7%) Cl(8.0%)

IR (KBr); [HL]: (cm^{-1}): 3800-2700 (-OH), 1599,1507,3028 (Aromatic), 1638, 1575,1698 (8-HQ Moiety), 1275-1298 (C-N), 2850,2920,1450 ($>\text{CH}_2$) ; PMR ; [HL]: δ ppm 7.1 to 7.64 Multiplet, quinoline, δ ppm 8.5 to 9.2 Singlet of phenolic- OH, δ ppm 4.75 - CH_2 -, δ ppm 1.75 - CH_3

Preparation of Metal Chelates of 2-(8-Quinololinol-5-yl) - amino methyl-3-(4- Methyl Phenyl)-5- (4-chloro Phenyl)-Pyrazoline. (2)

Formation of Co^{2+} Chelates :

It was obtained as mist colored precipitate by mixing a reagent solution of ligand (0.01 mole) with that of cobalt nitrate hexahydrate (0.005 mole) in 100 ml. of water. The final pH was adjusted 6.0. A brown complex was purified in the same manner described earlier. The yield of a purified complex was 62%.

Formation of Ni^{2+} Chelates :

A reagent solution of ligand (0.01 mole) mixed with a solution of nickel nitrate hexahydrate (0.005 mole) in 100 ml. of water. A dull greenish precipitate was formed at pH 6.0. The product was purified in the manner described above. The yield was 68 %.

Formation of Mn^{2+} Chelates :

The reagent solution of ligand (0.005 mole) was stirred in a solution of manganese chloride hexahydrate (0.005 mole) in 100 ml. of water. The final pH adjusted was 5.6. The yield of complex was 70%.

Characterization of Metal Chelates of Ligand

Metal Complexes	Molecular formula	M.W	Yield %	% Metal analysis		Elemental analysis					
						%C		%H		%N	
				Cald.	Found	Cald.	Found	Cald.	Found	Cald.	Found
$(\text{HL})_2 \text{Co}^{+2}$	$\text{C}_{52}\text{H}_{44}\text{N}_8\text{O}_2\text{Cl}_2 \text{Co}^{+2} \cdot 2\text{H}_2\text{O}$	978	62	6.0	5.9	63.8	63.7	4.9	4.8	11.4	11.4
$(\text{HL})_2 \text{Ni}^{+2}$	$\text{C}_{52}\text{H}_{44}\text{N}_8\text{O}_2\text{Cl}_2 \text{Ni}^{+2} \cdot 2\text{H}_2\text{O}$	978	68	6.0	5.9	63.8	63.7	4.9	4.8	11.4	11.4
$(\text{HL})_2 \text{Mn}^{+2}$	$\text{C}_{52}\text{H}_{44}\text{N}_8\text{O}_2\text{Cl}_2 \text{Mn}^{+2} \cdot 2\text{H}_2\text{O}$	974	70	5.6	5.6	64.0	63.9	4.9	4.8	11.5	11.4

IR (KBr); $(\text{HL})_2\text{-Mn}^{+2}$: (cm^{-1}): 3500-2600 broad (-OH), 1611,1461,2930 (Aromatic), 1650,1510, 1469 (8-HQ Moiety), 1281 (C-N), 2841,2930,1461 ($>\text{CH}_2$).

Experimental data of magnetic moment and conductivity of metal chelate of Ligand.

Metal complexes	$\chi_\gamma \times 10^6$ (cgs)	$\chi_\mu \times 10^6$ (cgs)	Magnetic moment μ_{eff} (BM)	$\mu_{\text{eff}} = \sqrt{n(n+2)}$ BM	μ_{eff} (BM) Expected	Λ_M^a
$(\text{HL})_2 \text{Co}^{+2}$	11.31	11057	5.18	3.87	4.4-5.2	24.10
$(\text{HL})_2 \text{Ni}^{+2}$	4.84	4736	3.39	2.82	2.9-3.4	8.10
$(\text{HL})_2 \text{Mn}^{+2}$	15.08	14687	5.97	5.91	5.2-6.0	10.02

Reflectance spectral data of metal complexes of ligand

Metal complex	Absorption, cm ⁻¹	Transional
(HL) ₂ Co ⁺²	23965	⁴ T _{1g} (F) → ⁴ T _{2g} (P)
	20115	⁴ T _{1g} (F) → ⁴ A _{2g}
	8749	⁴ T _{1g} (F) → ⁴ T _{2g} (F)
(HL) ₂ Ni ⁺²	23134	³ A _{2g} → ³ T _{1g} (P)
	15785	³ A _{2g} → ³ T _{1g} (F)
(HL) ₂ Mn ⁺²	22987	⁶ A _{1g} → ⁴ A _{1g} (4E _g)
	18970	⁶ A _{1g} → ⁴ T _{2g} (4G)
	15827	⁶ A _{1g} → ⁴ T _{1g} (4G)

Antifungal activity of ligand HL and their metal Chelate

Sample	Zone of inhibition at 1000 ppm (%)				
	Penicillium Expansum	C.Albicans	Nigras Pora Sp.	Trichothesium Sp.	A. Niger
HL	63	67	70	67	65
(HL) ₂ Co ⁺²	67	72	72	68	70
(HL) ₂ Ni ⁺²	62	65	60	60	67
(HL) ₂ Mn ⁺²	58	60	54	60	55

Antibacterial activity of ligands HL and their metal Chelate

Sample	Zone of inhibition (in mm)			
	Gram + Ve		Gram -Ve	
	B.Cereus	Micrococcus	P. Aeruginosa	E-Coli
HL	20	19	23	21
(HL) ₂ Co ⁺²	16	17	17	15
(HL) ₂ Ni ⁺²	14	12	15	14
(HL) ₂ Mn ⁺²	12	12	13	13

RESULT AND DISCUSSION

All the complexes are toxic more or less to fungi. The substitution of phenyl rings does not have more effect on the fungicidal activity of complexes. Out of all metal complexes, Co⁺² metal complexes are more toxic than others and the order for is Co⁺² > Ni⁺² > Mn⁺².

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