

Synthesis, Characterization and Biological Investigations on Metal Complexes Of 2-[3- Acetyl-2-Dihydroxybenzyl]-1h-Isoindole-1, 3(2h) – Dione

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Abstract

The more significant biologically active Cu (II), Ni (II), Co (II) and Mn (II) complexes were synthesized by using N-hydroxymethyl phthalimide and 2-hydroxyacetophenone as ligand. Synthesized complexes were characterized by elemental analysis, conductance, magnetic susceptibility, IR and reflectance spectral studies. Coordination of the ligand atom to the metal ion was deduced by IR and reflectance spectral data. Structures of the complexes were also confirmed by magnetic studies. All the complexes have been screened for antibacterial and antifungal activities. The substitution of phenyl rings does not have more effect on the fungicidal activity of chelates but in each series, the Cu-chelates have much toxicity. This is expected because the copper salts are mostly used as fungicides. Most of the compounds inhibit the growth of above organisms which cause disease in many plants.

Key Words: Hydroxyl methyl phthalimide, Antibacterial, Antifungal, 2-hydroxyacetophenone

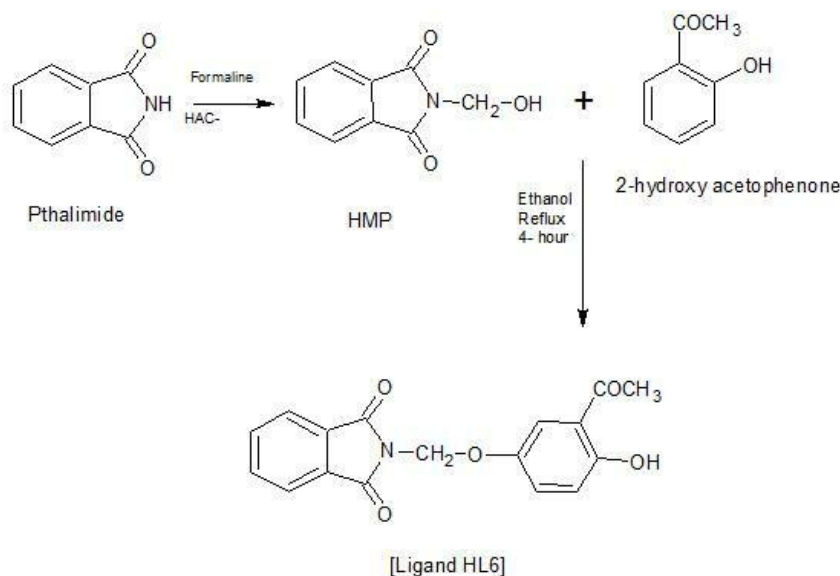
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I. INTRODUCTION

Survey of literature revealed the importance of N-aryl hydroxymethyl phthalimide derivatives as biologically, pharmacologically and industrially important molecules. The complexes obtained from the ligands having acetyl, benzyl, hydroxy and ketone functional groups have been investigated exhaustively.^{1 ref.}

Receiving impetus from the above observations and in continuation of our research programmed on complexes of HMP derivatives, in this paper, We report the synthesis of ligand 2-[3-acetyl-2-hydroxy benzyl]-1H-isoindole-1,3 (2H)- dione and its complexes with Cu (II), Ni (II), Mn (II) and Co (II) and also biological activities of these complexes. Hydroxymethyl phthalimide, a potential donor ligand with four coordination sites, is having the structure –^{2,3 ref.}



II. EXPERIMENTAL

Material and Methods

Phthalimide and the salts used for synthesis of novel phthalimide derivatives were obtained from the market. The metal salts and other chemicals used were of laboratory grade. Solvent were dried and distilled before used. The following metal salts were used to prepare complexes: copper acetate, cobalt acetate, nickel acetate and manganese acetate for the preparation of ligand 2- [3-acetyl-2-hydroxy benzyl]-1H-isoindole-1,3 (2H)-dione.

A solution of 0.04 mole 2-hydroxyacetophenone in ethanol and solution of 0.04 mole hydroxymethyl phthalimide (HMP) was taken in 50 mL ethanol and 5 drops of con. HCl were added. The two solutions were mixed with vigorous stirring at room temperature. The resultant mixture was refluxed for about 4 hours and cooled. The precipitates were separated, dried and crystallized with acetone. The yield was about 60% and M.P. = 135° C (uncorrected).

General Procedure

The metal ion complexes of ligand HL₆ with Cu²⁺, Mn²⁺, Co²⁺ and Ni²⁺ were prepared. The procedure is –

To solution of metal acetate (0.01M) in water (25 mL), a solution of ligand HL₆ (0.02M) in ethanol : water mixture (50 : 50 v/v) (100 mL) was added gradually with vigorous stirring at room temperature. The resultant mixture was refluxed for about 4 hrs. The solid complexes were separated by concentrating the resultant solution and adding ethanol : water mixture (70 : 30 ratio). The precipitates were filtered and washed several times with ethanol : water (70 : 30 ratio). The precipitates were dried at about 60°C. Yield was about 63 to 73%. The details of the elemental analysis, molar conductance of the complexes are given in Table 1.

Ligand HL₆ and its synthesized complexes were insoluble in water, slightly soluble in methanol, ether, chloroform, benzene and other organic solvents. They were soluble in DMF and acetonitrile. All the samples were analysed by elemental analyzer Thermo Finiggan 1101 Flash EA. Melting points were determined in open capillary and are uncorrected.

III. Analysis Discussion

Table 1: Physical and analytical data of ligand HL₆ and their metal complexes

Metal complex	Yiel d(%)	Mol. wt. (g/mole)	Elemental analysis						Metal %		Conductivit y Mohs.Cm ²
			C %		H%		N%				
			Cald.	Found.	Cald.	Found	Cald.	Found	Cald.	Found	
(HL ₆) ₂ Cu ⁺² 2H ₂ O	67	687.54	59.54	59.30	4.07	4.00	4.07	4.00	9.24	9.20	7.70
(HL ₆) ₂ Co ⁺² 2H ₂ O	63	682.94	59.74	59.70	4.09	4.00	4.09	4.00	8.63	8.60	9.60
(HL ₆) ₂ Ni ⁺² 2H ₂ O	70	682.69	59.76	59.70	4.10	4.10	4.10	4.10	8.59	8.50	8.70
(HL ₆) ₂ Zn ⁺² 2H ₂ O	68	689.38	59.18	59.10	4.06	4.00	4.06	4.00	9.48	9.40	11.70

The molar conductance of the complexes was measured using Elico CM-28. Magnetic susceptibility measurements were made on a Gouy balance at room temperature using conductivity water and mercury tetrathiocyanatocobaltate (II) [HgCo(CNS)₄] as calibrant. The reflectance spectra of the metal chelates in the visible region were recorded against MgO on Beckman-DK-2A spectrophotometer. The IR spectra of metal complexes were recorded in KBr in the range of 4000-400 cm⁻¹ Unicam FTIR (research spectrophotometer series) and in the range of 4000-200 cm⁻¹ were recorded on Perkin Elmer-577 IR spectrophotometer. The antimicrobial activities of the synthesized metal complexes were carried out by the cup-plate method. *In vitro* antimicrobial activity was carried out against 24 hrs old cultures of two bacteria, namely *Staphylococcus aureus* and *E.Coli* and 48 hrs old culture of two fungi, namely *Nigrospora Sp.* and *Rhizopus nigricum*.

Table 2: Experimental data of magnetic moment for metal chelates of HL₆

Metal chelates	$\chi_g \times 10^{-6}(\text{cgs})$	$\chi_m \times 10^{-6}(\text{cgs})$	Magnetic moment μ_{eff} (BM)	$\mu_{\text{eff}} = \sqrt{n(n+2)}$ BM obs.	$\mu_{\text{eff}} = \sqrt{n(n+2)}$ BM Expected
(HL ₆) ₂ Cu ⁺² 2H ₂ O	2.24	1541	1.89	1.73	1.7-2.2
(HL ₆) ₂ Co ⁺² 2H ₂ O	14.48	9889	4.81	3.87	4.4-5.2
(HL ₆) ₂ Ni ⁺² 2H ₂ O	6.13	4189	3.10	2.82	2.9-3.4
(HL ₆) ₂ Mn ⁺² 2H ₂ O	17.58	11936	5.10	5.91	5.2-6.0

The electronic spectrum of Co (II) complexes exhibited three bands around, 24875, 19230 and 8932 cm⁻¹, which are assigned to (V₁) ⁴T_{1g} (F) \square ⁴T_{2g}(F) , (V₂) ⁴T_{1g} (F) \square ⁴A_{2g} and (V₃) ⁴T_{1g} (F) \square ⁴T_{2g}(P), respectively in an octahedral environment field. The electronic spectrum of Ni (II) complex also showed three bands. In electronic spectra of Cu²⁺ metal chelate, the band may be assigned to ²B_{1g}→²A_{1g} and charge transfer transition, respectively. These results reveal the distorted octahedral geometry for these chelates.

The reflectance spectra of the metal chelates in the visible region were recorded against MgO on Beckman-DK-2A spectrophotometer. The assignment of significant bands present in the reflectance spectra of each of the metal chelates are given in Table 3.

Table 3: Reflectance spectral data of metal complexes of ligand HL₆

(HL ₆) ₂ — Cu ²⁺		22978 ⁶ A _{1g}	→	⁴ A _{1g} (4G)
		18929 ⁶ A _{1g}	→	⁴ T _{2g} (4G)
		15828 ⁶ A _{1g}	→	⁴ T _{1g} (4G)
(HL ₆) ₂ — Co ²⁺		23580 ⁴ T _{1g} (F)	→	⁴ T _{2g} (F)
		17413 ⁴ T _{1g} (F)	→	⁴ A _{2g}
		08692 ⁴ T _{1g} (F)	→	⁴ T _{2g} (P)
(HL ₆) ₂ —Ni ²⁺	23580	³ A _{2g}	→	³ T _{1g} (P)
		14940 ³ A _{2g}	→	³ T _{1g} (F)
(HL ₆) ₂ — Mn ²⁺	24522	Charge transfer	→	
		15622 ² B _{1g}	→	² A _{1g}

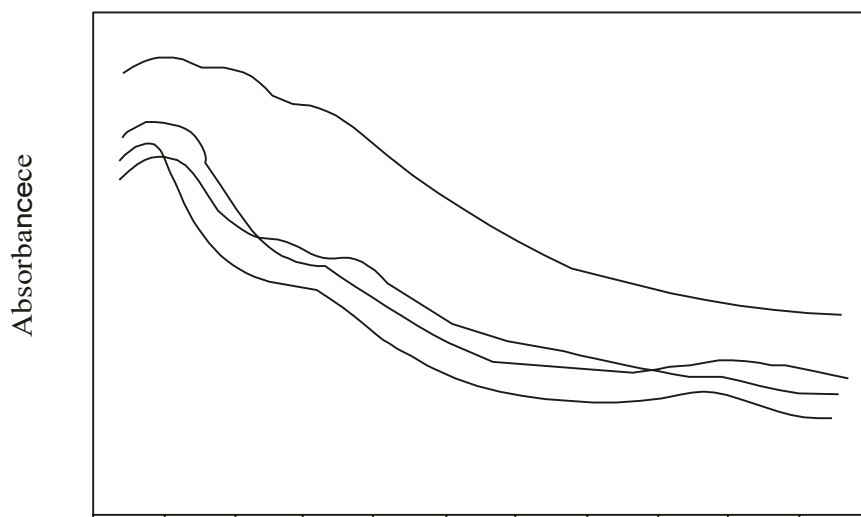


Fig. 1: Reflectance spectra of HL₆ metal complexes (1) HL₆-Cu (2) HL₆-Ni (3) HL₆-Co and (4) HL₆-Mn

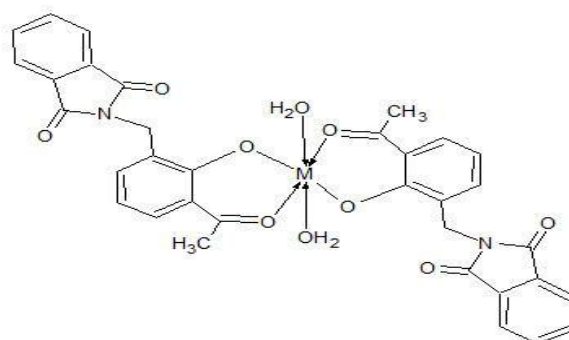
The band value in nm was converted into cm^{-1} and discussed. The only observable difference in the IR spectra of metal chelates and corresponding ligands has been the disappearance of the C=O stretching vibration in ligands at 1717 cm^{-1} on complex formation. A new band at 1095 cm^{-1} has appeared in the spectra of metal chelates.

Table 4: Infrared frequencies of HL₆ and their metal complexes

Frequency (cm^{-1})					
Ligand	Aromatic	Hydroxy -OH	Imides group	C=O ketone	-CH ₂ group(Bridge)
HL ₆	1597 C=C str.	3370 str.	1679 str.	1717 str.	2920 str. (Asym.)
	1498 C-H bend.		1580 str.	1685 str. -CH ₃	2850 str. (Sym.)
	3028 C-H str.	1381 bend.	1390 str.		1455 bend.
HL ₆ -M	1597 C=C str.	Metalligand 580-450	1679 str.	1717 str.	2920 str. (Asym.)
	1498 C-H bend.		1580 str.		2850 str. (Sym.)
	3028 C-H str.		1390 str.		1455 bend.

This may be assigned to $\nu_{\text{C-O}}$ of C-O-metal bond formation. The important bands are observed at their respective positions. The bands at 1679 cm^{-1} is due to imides group. The bands at 3028, 1597 and 820 cm^{-1} are mainly from 1,4-disubstituted aromatic ring where as the bands at 2920, 2850 and 1455 cm^{-1} are assigned to -CH₂ bridge system. The bands at 1685 cm^{-1} is due to -CH₃.

Fig. 2: Metal complexes of HL₆ M = Cu (II), Ni (II), Co (II) and Mn (II)



Metal Complexes of HL₆

Table 5: Antibacterial and antifungal activity of ligand HL₆ and their metal complexes

Sample	Zone of inhibition (in mm)		Zone of inhibition at 1000	
	<i>Staphylococcus Aureus</i>	<i>Ps. Aeruginosa</i>	<i>Penicillium Expansum</i>	<i>Nigrospora Sp.</i>
HL ₆	19	18	79	80
HL ₆ -Cu ⁺²	18	10	80	89
HL ₆ Mn ⁺²	14	12	83	81
HL ₆ -Co ⁺²	09	12	78	66
HL ₆ -Ni ⁺²	19	11	71	69

IV. RESULTS AND DISCUSSION

The data of electrical conductivity of all the complexes in acetone indicate that they are less polarizing in DMF. The magnetic moment of Co, Ni and Cu complexes were calculated from the corrected magnetic susceptibilities. The magnetic moment value of 2.82

B.M. was observed for complex of Ni (II), which is well within the range of 2.9-3.4 B.M. expected for octahedral complexes. The Cu (II) complex exhibited magnetic moment of the order of 1.73 B.M., which agrees well with the spin only value, suggesting an octahedral structure. The Mn (II) complex exhibited magnetic moment of the order of 5.91 B.M., which show highly paramagnetic nature.

The ligand, all the complexes synthesized in the present investigation and the respective metal salts were evaluated for antimicrobial activity.

Ligand and all the metal complexes were screened for their antibacterial activity against two bacterial strains viz. *Pseudomonas aeruginosa* and *Staphylococcus aureus* (Table 5). These compounds were also evaluated for their antifungal activity against two fungi viz, *Penicillium Expansum* and *Nigrospora Sp.*

The legends HL₆ were characterize with Elemental analysis, IR Spectral studies and Functional group determination. All the IR spectra are giving the important features of almost all aspects. All the metal chelate was examine with IR-Spectral analysis, Magnetic susceptibility, and Reflectance spectral study.

The complexes are toxic more or less fungi. The substitution of phenyl rings do not have more effect on the fungicidal activity of chelates but-OCH₃ more effect on the bactericidal activity

of complex. In each series the Cu-chelates have much toxicity. This is expected because the copper salts are mostly used as fungicides. Most of the compounds inhibit the growth of the above organisms which cause disease in many plants. Ligands HL₁ are more toxic because of the presence of -OCH₃ group. Dye prepared from salicylic acid is more toxic than all other Ligand because it is antifungal and antibacterial. Out of all metal chelates, Cu⁺² metal chelates is more toxic than others and the order for is

Cu⁺²>Zn⁺²>Co⁺²>Ni⁺²>Mn⁺²

V. CONCLUSION

The structures of all the complexes have been assigned based on various spectral and physical parameters. All the complexes possess octahedral geometry. Two water molecules are also involved in complexation. All the complexes exhibited considerable antibacterial activity against *staphylococcus aureus* and *pseudomonas aeruginosa* and anti-fungal activity against *Penicillium Expansum* and *Nigrospora Sp.*

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