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Synthesis Characterization and Antimicrobial Activity of Metal Chelates of 1, 7-Di (2-hydroxyphenyl) 1, 7 di (*N*-methyl) aza-1, 3, 5 heptarine-3-ol hydrochloride

NALIN P. PATANI* and M. V. HATHI

*C. U. Shah Science College, Ahmedabad-380014, Gujarat, India.

R. R. Mehta College of Science, Palanpur-385001, Gujarat, India.

nppatani@yahoo.co.in

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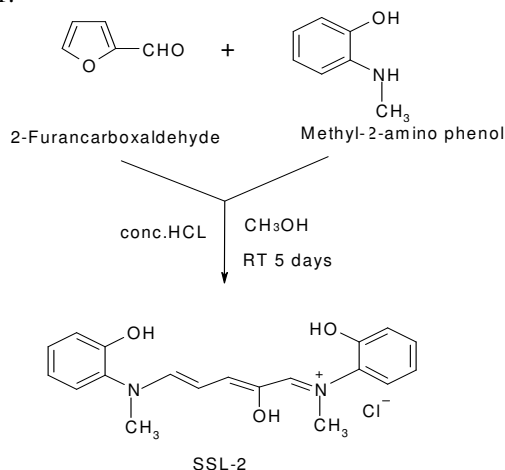
Abstract: The Schiff base complexes derived from 2-furancarboxaldehyde and *N*-methyl-2-amino phenol have been prepared and characterized using several physical techniques, like elemental analysis, thermogravimetric analysis, magnetic moment measurements, infrared and resonance spectra. *N*-Methyl-2-amino phenol was condensed stoichiometrically with 2-furancarboxaldehyde in presence of methanol. The resulting 1,7-di(2-hydroxyphenyl) 1,7 di(*N*-methyl) aza-1,3,5 heptarine-3-ol hydrochloride was characterized by elemental analysis and spectral studies. The transition metal complexes of Cu^{2+} , Zn^{2+} , Ni^{2+} , Co^{2+} and Mn^{2+} of the synthesized complexes were prepared. Metal ligand (M:L) ratio, IR, reflectance spectral studies, magnetic properties and antimicrobial activity of the synthesized complexes and its metal complexes were carried out.

Keywords: 2- Furancarboxaldehyde, *N*-Methyl-2-amino phenol, Metal complexes, Magnetic moment, Antifungal activities.

Introduction

2- Furancarboxaldehyde is well known as an analytical reagent^{1,2}. Its various derivatives³ are also useful. The complexes obtained from ligands having methyl, ethyl, nitro as functional group⁴ have been investigated exhaustively. Receiving impetus from the above observation and continuation of our research program, it was thought to undertake such view. The present communication describes the synthesis and characterization of 1,7-di(2-hydroxyphenyl) 1,7 di(*N*-methyl)aza-1,3,5 heptarine-3-ol hydrochloride and its complexes

with Cu^{2+} , Zn^{2+} , Ni^{2+} and Co^{2+} also antimicrobial activities of these complexes. This work is illustrated in Scheme 1.



Scheme 1

Experimental

2-Amino phenol was obtained from local dealer in market. The metal salts and other chemicals used were of analytical grade. To a solution of (0.1 M) *N*-methyl-2-amino phenol in methanol, a solution of 2- furancarboxaldehyde (0.05 M) in methanol was added drop wise within 20 minutes. Then 9 mL conc. HCl was added slowly with maintaining the room temperature. The mixture was kept aside for five days with occasional stirring. The resulting dark violet compound was filtered and washed by methanol and then with ether⁵. Finally it was air-dried. The yield was about 63%. The Cu^{2+} , Zn^{2+} , Ni^{2+} and Co^{2+} metal ion complexes of ligand SSL-2 were prepared using the following procedure.

The solution of 25 mL disodium salt containing 0.01 M ligand was taken, then each ligand was added drop wise to a solution of 0.005 M cupric nitrate hexahydrate in 100 of water with rapid stirring. The pH of the resultant solution was 4.5. The solid mass was precipitated out. It was allowed to settle down then digested on water bath at 70 °C for about 2 h. The solid mass was filtered, washed with 1:1 mixture of water-ethanol and finally with acetone and air-dried, the resulting yield was 70%. The resulting complex was powdered well and further dried at 70 °C over a period of 24 h. The details of the elemental analysis of the complexes are given in Table 1. Ligand 1,7-di(2-hydroxyphenyl)-1,7-di(*N*-methyl)aza-1,3,5 heptatriene-3- ol-hydrochloride (SSL-2) and its synthesized complexes were insoluble in water, slightly soluble in methanol, ether, chloroform and other organic solvents. They were soluble in DMF. All the samples were measured by elemental analyzer Thermo Finiggan 1101 Flash EA⁶. IR spectra of SSL-2 and its metal complexes were scanned on Nicolet 760 FTIR Spectrophotometer in KBr. The metal content of the metal chelates by decomposing a weighed amount of each metal complexes were analysed followed by EDTA titration as reported in literature⁷. Magnetic susceptibility measurement of all metal complexes was carried out at room temperature by the Gouy method⁸. The values are given in Table 2. Mercury tetrathiocyanatocobaltate(II) was used as a calibrant⁹. The reflectance spectra of the metal chelates in the visible region were recorded against MgO on Backman DK 2A Spectrophotometer (Figure 1). The antimicrobial activities of the synthesized metal complexes were carried out by cup-plate method¹⁰. All the samples were monitored against various fungi following the method reported in literature¹¹.

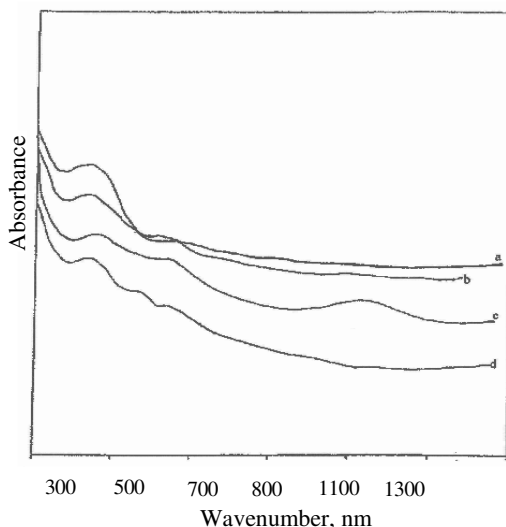


Figure 1. Reflectance spectra of SSL-2 metal complexes (1) SSL-2 Cu²⁺ (2) SSL-2 Ni²⁺ (3) SSL-2 Co²⁺ (4) SSL-2 Mn²⁺.

Results and Discussion

1,7 Di(*N*-methyl)aza-1,3,5 heptarine-3-ol hydrochloride was dark violet powder. The result of elemental analysis of ligand is reported in Table 1. The values are consistent with the predicted structure (Scheme 1). The IR spectra of 1,7 di(*N*-methyl)aza-1,3,5 heptarine-3-ol hydrochloride comprises the important bands¹² observed at 3600 to 2600 cm⁻¹. The IR spectra of all the chelates suggest that the formation of entire metalocyclic compound by the absence of band characteristic of free -OH group. The electronic spectrum of Co(II) complexes exhibits three bands around 22834, 16462 and 9222 cm⁻¹. Which are assigned ${}^6A_{1g}(F) \rightarrow {}^4T_{2g}(F)$, ${}^6A_{1g}(F) \rightarrow {}^4T_{2g}$, ${}^6A_{1g}(F) \rightarrow {}^4T_{2g}(P)$ respectively in octahedral field¹³. The magnetic moment¹⁴ of the order is 3.76 B.M. The electronic spectra of Cu²⁺ metal chelate of each of the ligand may be assigned to ${}^2B_{1g} \rightarrow {}^2A_{1g}$ and charge transfer transition respectively. The electronic spectral of Mn²⁺ complexes were also show three bands. The examination of antifungal activity¹⁵ of SSL-2 ligands and its all chelates (Table 3) indicates that all compounds are good toxic for fungi. Out of all the compounds copper chelates is more than that of other (Table 3).

Table 1. Analytical data of the metal chelates of SSL - 2.

Molecular formula	Yield %	Mol. Wt.	Elemental analysis						Metal, %	
			C %		H %		N %		Cald.	Found
			Cald.	Found	Cald.	Found	Cald.	Found		
C ₁₉ H ₂₀ N ₂ O ₄ Cu ²⁺ ·2H ₂ O	65	439.5	51.8	51.6	4.6	4.5	6.4	6.3	14.4	14.3
C ₁₉ H ₂₀ N ₂ O ₄ Mn ²⁺ ·2H ₂ O	67	431	52.9	52.7	4.6	4.5	6.5	6.4	12.7	12.6
C ₁₉ H ₂₀ N ₂ O ₄ Ni ²⁺ ·2H ₂ O	57	435	52.4	52.3	4.6	4.5	6.4	6.3	13.5	13.4
C ₁₉ H ₂₀ N ₂ O ₄ Co ²⁺ ·2H ₂ O	62	435	52.4	52.4	4.6	4.5	6.4	6.3	13.5	13.4
C ₁₉ H ₂₀ N ₂ O ₄ Zn ²⁺ ·2H ₂ O	60	441	51.7	51.6	4.5	4.5	6.3	6.3	14.7	14.6

Table 2. Experimental data of magnetic moment of metal chelates of SSL- 2.

Metal Complex	$\chi_g \times 10^6$ (cgs)	$\chi_m \times 10^6$ (cgs)	Observed Magnetic moment μ_{eff} (B.M.)	Theoretical $\mu_{\text{eff}} = \sqrt{n(n+2)}$ (B.M.)
(SSL-2) Cu ²⁺	2.55	1121.89	1.65	1.73
(SSL-2) Ni ²⁺	7.42	3230.72	2.80	2.82
(SSL-2) Co ²⁺	13.39	5825.86	3.76	3.87
(SSL-2) Mn ²⁺	33.84	14588.76	5.95	5.91
(SSL-2) Zn ²⁺	-	-	-	D*

Table 3. Antifungal activity of SSL-2 and their metal complexes.

Sample	Zone of inhibition at 1000 ppm, %				
	<i>Penicillium Expansum</i>	<i>F. Udum</i>	<i>Nigras Pora Sp.</i>	<i>Trichoderma Sp.</i>	<i>Erysiphe pisi</i>
SSL-2	54	55	54	62	65
(SSL-2) ₂ Cu ²⁺	88	78	82	88	75
(SSL-2) ₂ Mn ²⁺	62	70	74	68	75
(SSL-2) ₂ Zn ²⁺	75	70	66	74	66
(SSL-2) ₂ Co ²⁺	66	54	70	69	66
(SSL-2) ₂ Ni ²⁺	54	58	78	45	64

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