

Synthesis, characterization of antimicrobial activity of Fe(III) complexes of benzoinhydrzones

P.M.Dahikar¹, R.M Kedar²

¹Shr .R.R Lahoti Science College Morshi, Dist. Amravati

²Shri. Shivaji Science College Amravati.

ABSTRACT

Metal Benzoinhydrazone and complexes have been synthesized from substituted benzoinhydrazones. They were characterized by elemental and spectral analysis. The synthesized complexes were screened for antimicrobial activity at a concentration of 1000µ gm/ml which was serially diluted to determine their MIC value of benzoinhydrazone-Cu(II).

Keywords – Antimicrobial activity Fe(III) complexes, 4-Dimethylamino benzoinhydrazone, 4,4'-dimethoxybenzoinhydrazone, 2-hydroxybenzoinhydrazone and furionhydrazone.

1. INTRODUCTION

Benzoinhydrazone are well known for their bacterial activity coordination compounds containing donor atoms are reported to possess antimicrobial activity¹. Shcherbakov² carried out specificity of complex formation of Benzoin (Phthalazin-1-Yl) hydrazone with copper (II) and Nickel (II). A series of quinoxalinone derivatives were synthesized and studied their antimicrobial and antiinflammatory activities were studied by Khan³ synthesise of o-amino-acetophenone o-hydroxybenzolyhydrazone. Complexes of divalent transition metals and studied their spectral and antimicrobial activities was reported by Nawar⁴. A structural analysis of salicylaldehyde benzoyl hyarazone by using a combination of NMR, IR and theoretical investigation was carried but by cordier⁵.The simultaneous spectroscopic determination of palladium and osmium with salicylaldehyde hydrazone was carried out by Ray⁶.The synthesis and structural characterization of three new co-ordination complexes of Co (II), Mn (II) nad Cu (II) with N,N,O-donor hydrazine lignds were carried out by shit⁷. The synthesis, magnetic, spectral, thermal and biological studies of Ti(III), Vo (IV), Cr (III), Mn (II), Fe (III) and Zr (IV) Complexes with chelating hydrazine derived from 2-hydroxy-5 methylacetophenone and furoic acid hydrazide carried out by Dhande⁸. Thermal analysis in structural characterization of hydrazone ligands and their complexes studied by Andyelkovic⁹. The synthesis of novel (II) complexes with Now ligand derived from hydrazone of isoniazid and their magnto-spectral, electrochemical, thermal and antimicrobial investigations were studied by Prasad¹⁰.

2. EXPERIMENTAL

The benzoinhydrazone were prepared by refluxing substiltuted benzoin with hydrazine hydrate in presence of alkaline medium for 3-4 hours. Reaction mixture were kept overnight. The solid products formed were isolated and washed several times with water alcohol mixture the purity was checked by TLC paper. Their structural details were confirmed on the basis of elemental and spectral analysis.

In order to synthesize the complexes the equimolar mixture of each of the ligand (0.01 M) and metal salts (0.01) were refluxed on a water bath for 6,8 hours in presence of sodium acetate in ethanol / methanol. The reaction mixture was kept overnight. The products formed were isolated washed several times with cold water-ethanol mixture. The characterization of synthesized complexes was made with elemental analysis, IR and UV-VIS spectra.

3. PHYSICAL MEASUREMENTS

C, H, and N were analysed on a carlo-Erba 1106 elemental analyser. Magnetic susceptibility was measured at room temperature on Gouy balance using Hg [Co(SCN)₄]₂ as calibrant. ¹HHMR spectra were recorded on Bruker AC 300F spectrometer with TMS as internal standard using CDCl₃ and DMSO-D₆ as a Solvent. IR spectra (KBr) were recorded on perkin Elmer spectrometer in range 4000-400cm⁻¹ in KBr pellets. All chemicals used were of AR-grade.

4. RESULT AND DISCUSSION

On the basis of elemental analysis the complexes were assigned the composition as shown in table. 1

Complexes	M.Wt.	Colours	Elemental analysis Found / (Calculated) %			
			C	H	N	M
4-DMABH-Mn(II)	630.93	Greenish	59.85 (60.86)	4.81 (5.70)	14.11 (14.11)	14.01 (15.04)
4,4'-DMBH-Mn(II)	666.93	Greenish	56.58 (57.57)	4.55 (5.39)	8.39 (8.39)	13.37 (14.23)
FUROH-Mn(II)	388.93	Gray	60.81 (61.70)	3.73 (4.62)	6.26 (7.19)	13.27 (14.12)
4-DMABH-Fe(III)	591.84	Redbrown	63.93 (64.88)	5.03 (6.08)	13.31 (13.31)	8.66 (9.43)
4,4'-DMBH-Fe(III)	627.84	Brown	60.24 (61.16)	4.86 (5.73)	8.91 (8.91)	7.96 (8.89)
FUROH-Fe(III)	389.84	Brown	60.73 (61.56)	3.70 (4.61)	6.24 (7.18)	13.42 (14.32)

Table 2 Magnetic moment and spectral data of the metal complexes.

Complexes	μ_{eff} (B.M.)	$\lambda_{\text{max}}(\text{cm}^{-1})$	Dq	B^1	B	%cova
4-DMABH-Mn(II)	5.79	13769,18098,21113	1490	565	0.588	42
4,4'-DMBH-Mn(II)	5.78	14076,19917,21375	1521	562	0.586	41.4
FUROH-Mn(II)	5.89	13870,19194,21739	1506	603	0.629	37.1
4-DMABH-Fe(III)	5.63	14161,20213,24352	1556	772	0.761	23.9
4,4'-DMBH- Fe (III)	5.67	13888,17979,22471	1514	655	0.646	35.4
FUROH-Fe (III)	5.65	13698,18518,22222	1494	651	0.641	35.9

4-DMABH-Mn(II) complex showing bands near 13769, 18098, 21113 cm^{-1} respectively may be assigned to the transition which may be due to ${}^6A_{1g}(S) \rightarrow {}^4T_{1g}(G)$, ${}^6A_{1g} \rightarrow {}^4T_{2g}(G)$ and ${}^6A_{1g}(S) \rightarrow {}^4E_g, {}^4A_{1g}(G)$ transitions respectively octahedral geometry around Mn(II) ion the magnetic moment of 5.79B.M. Mn(II) complex suggest on octahedral geometry⁽¹¹⁻¹²⁾ 4-DMABH-Fe(III) complexes showing these bands are observed in case of Fe(III) complex at 14161, 20213, 24352 cm^{-1} belongs to ${}^6A_{1g} \rightarrow {}^4T_{1g}$, ${}^6T_{1g} \rightarrow {}^6A_{1g}$ and ${}^6A_{1g} \rightarrow {}^4E_g, {}^4A_{1g}$ transition respectively, indicating octahedral geometry of Fe(III) complex¹³. The value of 5.63 B.M. would suggest high spin six coordination for Fe (III) complexes¹⁴.

Table-3 IR spectral data of ligands and its metal complexes

Ligands and its complexes	$\nu(\text{O-H})$	$\nu(>\text{C}=\text{N})$	$\nu(\text{C-O})$	$\nu(\text{M-O})$	$\nu(\text{M-N})$
4-DMABH	3423	1597	1388	-	-
4,4'-DMBH	3388	1634	1362	-	-
FUROH	3420	1620	1419	-	-
4-DMABH-Mn(II)	3392	1549	1382	512	575
4,4' DMBH-Mn(II)	3340	1613	1301	450	576
FUROH-Mn(II)	3396	1587	1369	503	588
4-DMABH-Fe(III)	3414	1545	1376	489	566
4,4'-DMBH- Fe(III)	3378	1610	1249	471	565
FUROH-Fe(III)	3391	1581	1336	485	587

IR spectra of ligand 4-DMABH show band at 3423 cm^{-1} $\nu(\text{O-H})$ IR spectra of 4-DMABH-Mn(II) complexes show band which decrease to 3392 cm^{-1} indicating through hydrogen oxygen. However 1597($\text{C}=\text{N}$) significantly decreases to 1549 cm^{-1} showing linkage through imino nitrogen. IR spectra of ligands 4,4'-DMBH show band at 3388 cm^{-1} $\nu(\text{O-H})$.In 4,4'-DMBH Mn (II) Complexes

Which decrease to 3340cm^{-1} indicating through hydrogen oxygen. However $1634(\text{C}=\text{N})$ significantly decreases to 1613cm^{-1} showing linkage through imino nitrogen. IR spectra of ligands FURO show band at 3420cm^{-1} in FURO-Mn(II) complexes to which decreases 3396cm^{-1} indicating through hydrogen oxygen. However $1620(\text{C}=\text{N})$ significantly decreases to 1587cm^{-1} .

5. ANTIMICROBIAL ACTIVITY OF COMPLEXES

The compounds were assayed for their antimicrobial activities¹⁵. Against four test organisms. E. coli, S. aureus, P. aeruginosa, B. Subtilis at a concentration of $1000\text{ }\mu\text{g}/\text{ml}$ by agar well technique¹⁶, Further their MIC value against these organisms were determined by serial dilution method using DMSO as a solvent. The results obtained are given in

Table - 4

Complexes	E.coli	S.aureus	P. aeruginosa	B.Subtilis
4-DMABH-Mn(II)	125	125	125	63
4,4'-DMBH-Mn(II)	125	250	250	250
FUROH-Mn(II)	125	125	125	63
4-DMABH-Fe(III)	63	125	125	125
4,4'-DMBH-Fe(III)	125	250	125	125
FUROH-Fe(III)	125	125	63	63

On the basis of MIC values, FUROH-Fe(III) is found to be most effective antimicrobial agent followed by 4-DMABH-Mn(II) and 4-DMABH-Fe(III). The enhanced antimicrobial activity in case of the compound, FURO-Fe(III) may be attributed to the presence of furanyl group.

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