

Synthesis and Antimicrobial Study of Fe (III) and Cr (III) Complexes of Substituted Benzoinsemicarbazones

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Abstract: Metal benzoinsemicarbazone complexes were synthesized from substituted benzoinsemicarbazone. They were characterized by elemental and spectral analysis. The Physico-Chemical data suggest octahedral geometry for Cr(III) & Fe(III) complexes. The synthesized complexes were screened for antimicrobial activity at a concentration of 1000 µg/ml. Which was serially diluted to determine their MIC values.

Keywords: Metal complex, 4-Dimethylaminobenzoinsemicarbazone, Antimicrobial Study, Furionsemicarbazone, spectral analysis.

1. Introduction

The spectroscopic and biochemical studies of chromium and Mn(II) with p-vanillin containing thiosemicarbazone and semicarbazone ligands reported by chandra¹. Benzoinsemicarbazone are well known for their biological activity. Coordination compounds containing ONS as donor atoms are reported to possess antimicrobial activity². Kumar³ carried out synthesis and characterization of Mn(II), Fe(III), Co(II), Ni(II) and Cu(II) complexes of salicylaldehydesemicarbazone. Khan⁴ reported synthesis and characterization of Co(II), Ni(II), Cu(II) and Cd(II) complexes with 2-furfuralsemicarbazone (FSC) and 5-methyl-2-furfuralsemicarbazone (MFSC). Physico-chemical and spectral studies of Ni(II) complexes of 2-substituted benzaldehydesemicarbazone, and thiosemicarbazones were carried out by Kumar⁵. O-vanillin semicarbazone have been synthesized and characterized by different physicochemical techniques by Hingorani⁶, Choudhary⁷ carried out synthesis and characterization of new series of mixed ligand complex of Co(II) and Cu(II) with thiosemicarbazone/ Semicarbazone and screened their antibacterial and antifungal activities in detail. Investigation on variety of semicarbazones and Schiff bases and their transition metal complexes was carried out by several workers⁸⁻⁹. Mohapatra¹⁰ reported the complexes of divalent Mn(II), Co(II), Cu(II) with benzil semicarbazone. In the present work novel transition metal complexes of substituted benzoinsemicarbazone are reported. The magnetic moment values of Cr(III), Mn(II) and Fe(III) complexes are in good agreement with the presence of three, four and five unpaired electrons respectively¹¹.

2. Experimental Section

The benzoinsemicarbazone prepared by refluxing substituted benzoin with semicarbazide hydrochloride in presence of alkaline medium for 3-4 hours. Reaction mixture was 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 was refluxed on a water bath for 6 - 8 hrs 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 mixtures. 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 analyses. Molar conductance was measured on the conductometer EQ-660A. Magnetic susceptibility was measured at room temperature on a Gouy balance using Hg [Co(SCN)₄] as calibrant. ¹H NMR spectra were recorded on Bruker AC 300 F 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- 400 cm⁻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	Colour	Molecular wt	Elemental analysis Found/(calculated)%			
			C	H	N	M
4-DMABSC-Cr(III)	Brown	709.99	56.55 (57.46)	4.00 (5.91)	15.77 (15.65)	6.40 (7.32)
4-DMABSC-Fe(III)	Red	619.99	44.65 (45.58)	3.60 (4.51)	13.54 (13.45)	7.46 (8.38)
FUROS-Cr(III)	Brown	713.84	56.23 (57.15)	4.94 (5.88)	15.68 (15.48)	6.91 (7.82)
FUROS-Fe(III)	Grey	587.84	43.98 (44.91)	3.14 (4.08)	14.28 (14.18)	8.55 (9.49)

Table 2: IR spectral data of ligands and its metal complexes

Ligands and its Complexes	$\nu(O-H)$	$\nu(C=N)$	$\nu(C-O)$	$\nu(M-O)$	$\nu(M-N)$
4-DMABSC	3463	1569	1181	-	-
4-DMABSC-Cr(III)	3411	1549	1169	462	581
4-DMABSC-Fe(III)	3419	1545	1171	469	587
FUROSC	3433	1599	1152	-	-
FUROSC- Cr(III)	3399	1579	1128	465	583
FUROSC- Fe(III)	3397	1507	1129	469	586

IR spectra of 4-DABSC - Fe (III) shows band at 3463 (O - H) which decreases at 3411 cm⁻¹ indicating that hydrogen attached to oxygen. However 1569 (C=N) significantly decrease to 1545 cm⁻¹ showing linkage through azido nitrogen.

Table 3: Magnetic moment and spectral data of the metal complexes

Complexes	μ_{eff} (B.M.)	λ max (cm ⁻¹)
4-DMABSC-Cr(III)	4.07	13476,19590,22620
4-DMABSC-Fe(III)	4.16	13769,19794,22150
FUROSC- Cr(III)	5.81	14077,18565,23218
FUROSC- Fe(III)	5.83	13793,18518,22471

electronic spectrum of Cr(III) complexes exhibits three bands at 13476,19590 and 22620cm⁻¹ which may be assigned to ${}^4A_{2g} \rightarrow {}^4T_{2g}(F)$, ${}^4A_{2g} \rightarrow {}^4T_{1g}(F)$ and ${}^4A_{2g} \rightarrow {}^4T_{1g}(P)$, transition, respectively for an octahedral stereochemistry¹¹. The magnetic moment of 3.97 to 4.18 B.M for Cr(III) complex is consistent with octahedral geometry around metal centre Fe (III) Complexes . Three bands are observed in case of Fe(III) complexes at 14077, 18565, 23218 cm⁻¹ belongs to transition respectively, indicating octahedral geometry of Fe(III) complexes¹²⁻¹³. The value of 5.85 B.M. would suggest high spin six coordination for Fe(III) complexes.

5. Antimicrobial Activity of Complexes

The compounds were assayed for their antimicrobial activities¹⁴ against four test organisms E.coli, S. aureus, Ps.aeruginosa, B. subtilis at a concentration of 1000µg/ml by agar well technique¹⁵. Further their MIC value against these organisms were determined by serial dilution method using DMF as a solvent. The results obtained are given in table-4

MIC values in µg/ml of compounds

Complexes	E.coli	S.aureus	P. aeruginosa	B.Subtilis
4-DMABSC-Cr(III)	250	125	63	125
4-DMABSC-Fe(III)	125	125	250	250
FUROSC- Cr(III)	63	125	125	250
FUROSC- Fe(III)	250	250	125	250

On the basis of MIC values, FUROSC-Cr(III) is found to be most effective antimicrobial agent followed by 4-DMABSC-Cr(III) and 4-DMABSC-Fe(III). The enhance antimicrobial activity in case of the compounds. FUROSC-Cr(III) may be attributed to the presence of furanyl group.

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