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Synthesis, spectral and biological activity of transition metal complexes of substituted benzoinsemicarbazones

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ABSTRACT

Novel metal benzoinsemicarbazone complexes have been synthesized from substituted benzoinsemicarbazones. They are characterized by elemental and spectral analysis. The synthesized complexes were screened for antimicrobial activity at a concentration of 1000 µgm/ml which was serially diluted to determine their MIC values.

Keywords: Metal complexes 44'-Dimethoxybenzoinsemicarbazone, furionsemicarbazone, Antimicrobial activity.

1. INTRODUCTION

Benzoinsemicarbazone are well known for their biological activity coordination compounds containing ONS as donor atoms are reported to possess antimicrobial activity[1]. Kumar[2] carried out synthesis and characterizatio of Mn(II), Fe(III), Co (II), Ni (II) and Cu(II) complexes of salicyaldehydesemicarbazone . Khan[3] 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 Kumar[4]. Synthesis of mixed ligand CO(II) complexes with salicyladehyde, semi,thiosemi and isothiosemicarbazone and pyridine was carried out by Leovac[5]. Shaikh[6], selected vanillin semicarbazone (VSC) as a test compound and studied it's anticancer activity against EAC cell in vivo Chandra[7], reported synthesis and characterization of Co(II) complexes with semicarbazones and thiosemicarbazone and also t screened them for biological activities against E.coli, S.aureus, microorganisms. Siji[8], carried out FT- IR and FT - Raman spectral studies and DET calculation of tautomeric forms of benzaldehyde - N (4) phenyl - semicarbazone. The synthesis ,structural and spectral studies of di - 2 - pyridyl ketone N(4) - methyl and N(4) - dimethyl semicarbazone with Co (II), Ni (II), Cu(II) complexes were carried out by 5 wearingen and west[9].Agrawal Prasad reported the synthesis, spectral and thermal studies of dioxowranium (VI) with semicarbazones. Milkhaleva[10], carried out synthesized metal complexes of semicarbazones, thiosemicarbazones, guanylhydrazones of 1-vinyl - pyrrole - 2 - carbaldehydes and reported that these compounds have their own importance in parmaceutical and medicinal fields.Chandra[12] reported the spectroscopic and biochemical studies of chromium and Mn(II) with p-vanillin containing thiosemicarbazone and semicarbazone ligands.

2. EXPERIMENTAL SECTION

The benzoinsemicarbazone derivarities were prepared by refluxing substituted benzoins with semicarbazide hydrochloride in presence of alkaline medium for 3-4 hour's 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 was refluxed on a water both 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 conductormeter EQ-660A. Magnetic susceptibility was measured at room temperature on a Gouy balance using Hg [Co $(SCN)_4$] as calibrant. ^{1}HNMR spectra were recorded on Brucker AC 300 F spectrometer with TMS as internal standard using $CDCl_3$ and DMSO-D_6 as a solvent. IR spectra (KBr) were recorded on perkin Elmer spectrometer in range 4000 - 400 cm^- in KBr pellets. All chemicals used were of AR - grade

4. RESULTS AND DISCUSSION

On the basis of elemental analysis, the complexes were assigned to posses the composition as shown in Table 1.

Table 1: Elemental Analysis data found / (Calculated).

Complexes	C%	H%	N%	M%
4-DABSC - Co (II)	59.18 (60.27)	4.32 (5.02)	16.54 (16.54)	7.63 (8.70)
4-DABSC - Fe(II)	59.32 (60.54)	5.04 (4.94)	15.18 (16.62)	7.39 (8.28)
44'-ADMBSC - Co (II)	56.14 (57.04)	4.24 (5.03)	11.74 (11.74)	7.39 (8.24)
44'-DMBSC - Fe (III)	56.44 (57.31)	4.3 (5.0)	11.80 (11.80)	6.90 (7.84)
O-HBSC - Co(II)	56.49 (57.42)	3.59 (4.46)	13.39 (13.39)	8.52 (9.39)
O-HBSC - Fe (III)	56.82 (57.70)	5.65 (4.48)	13.46 (13.46)	7.89 (8.95)
FURSC - Co (II)	52.83 (53.77)	3.39 (4.07)	17.11 (17.11)	11.03 (12.00)
FURSC - Fe (III)	53.24 (54.11)	3.1 (4.09)	17.21 (17.21)	10.66 (11.44)

5. IR - SPECTRA OF COMPLEXES

In the spectra of ligands 4 - DABSC - Co (11) shows band at 3463 (O - H str) which decreases to3419 cm^{-1} indicating through hydrogen oxygen.However 1569 (C = N) significantly decreases to 1547 cm^{-1} showing linkage through azido nitrogen. IR spectra of P-DABSC - Fe (III) shows band at 3463 (O - H) which decreases at 3411 cm^{-1} inducating that hydrogen attached to oxygen. However 1569 (C=N) significantly decrease to 1545 cm^{-1} showing linkage through azido nitrogen.

Table -2

Complexes	Ω -1 cm2 mol-1	Colour	Decomp Temp(0C)	IR Key Band cm-1
4-DABSC-Co(II)	11.4	Yellow	280^0C	3419 (O-H),1547(C=N)
4-DABSC-Fe(III)	8	Red	333^0c	3411(O-H),1545(C=N)
44'-DMBSC-Co(II)	10.5	Dark brown	536^0C	3465(O-H),1656(C=N)
44'-DMBSC-Fe(III)	9.2	Red	536^0C	3397(O-H),1526(C=N)
O-HBSC-Co(II)	12.5	Black	285^0C	3397(O-H),1599(C=N)
O-HBSC-Fe(III)	10.5	Brown	336^0C	3398(O-H),1575(C=N)
FURSC-Co(II)	8.8	Black	291^0C	3384(O-H),1541(C=N)
FURSC-Fe(III)	9.8	Red	328^0C	3397(O-H)1507(C=N)

The molar conductance data 8-12.2 $\Omega^{-1}cm^2mol^{-1}$of all the complexes in DMF or DMSO indicates that these are non-electrolyte in nature.

6. AGNETIC MOMENT AND ELECTRONIC SPECTRA

Table -3

Complexes	μeff (B.M.)	λ max (cm^{-1})	Dq	B'	β	% Covalency
4-DABSC - Co(II)	5.15	21276, 17927, 13105	1421	562	0.59	42.04
4-DABSC - Fe(III)	5.79	23228, 18565, 14077	1538	697	0.687	31.27
44'-DMBSC - Co(II)	5.31	20408, 17843, 13063	1417	563	0.580	41.91
44'-DMBSC - Fe(III)	5.85	21957, 17833, 13422	1465	651	0.641	35.83
OHBSC - Co(II)	5.13	22471, 19417, 13605	1487	675	0.695	30.40
OHBSC - Fe(III)	5.70	22222, 19230, 13605	1485	657	0.647	35.20
FURSC - Co (II)	5.25	20616, 17953, 13087	1421	577	0.594	40.53
FUSRC - Fe (III)	5.72	22471, 18518, 13793	1505	662	0.652	34.7

Co (II) Complexes

Cobalt (II) complexes exhibits absorption bands at 13105 , 17927 and 21276 cm^{-1} which may be assigned to $^4T_{1g}(F) \rightarrow {}^4T_{2(g)}$, $^4T_{1g} \rightarrow {}^4A_{2g}$ and $^4T_{1g} \rightarrow {}^4T_{1g}(p)$ transitions respectively [13-14] suggesting an octahedral geometry around a cobalt ion, in the complexes under study. Furthermore, the magnetic moment measurements recorded at room temperature of an octahedral geometry [15-16] of these complexe.

Fe (III) Complexes

Three bands are observed in case of Fe(III) complexes at 14077, 18565, 23228 cm^{-1} belongs to $^6A_{1g} \rightarrow {}^4T_{1g}$, $^6A_1 \rightarrow {}^4T_{2g}$ and $^6A_{1g} \rightarrow {}^4E_g$, $^4A_{1g}$ transition, respectively, indicating octahedral geometry of Fe(III) complexes [17-18]. The value of 5.85 B.M. would suggest high spin six coordination for Fe(III) complexes[16]

7. ANTIMICROBIAL ACTIVITY OF COMPLEXES

The compounds were assayed for their antimicrobial activities[20] against four test organisms E.coli, S. aureus, Ps. aeruginosa, B. subtilis at a concentration of 1000μgm/ml by agar well technique[21]. Further their MIC value against these organisms were determined by serial dilution method[20] using DMF as a solvent. The results obtained are given in Table.

Table-4

Complex	E.Coli	S. aureus	Ps. Aeruginosa	B.Subtilis
4-DABSC-Co(II)	250	125	125	125
4-DABSC-Fe(III)	125	125	250	250
4-4'-DMBSC	250	125	250	250
4-4'DMBSC-Fe(III)	250	250	125	125
O-HBSC-Co(II)	125	250	125	125
O-HBSC-Fe (III)	250	250	125	250
FSC - Co (II)	250	125	63	63
FSC - Fe (III)	250	250	125	250

MIC Values μgm / ml of compounds.

8. RESULT AND DISCUSSION

On the basis of MIC values ,Furionsemicarbazone Co(II), is found is found to be most effective antimicrobialagent followed by O-hydroxybenzoinsemicarbazone Co(II) and 4-DimethylaminobenzoinsemicarbozoneCo(II),Similary 4-Dimethylaminobenzoinsemicarbazone-Fe(III) and 44'-Dimethmuchoxybenzoinsemicarbazone Fe(III) show much lower MIC values. However,former is more effective against E.coli and S.aureus. where as latter is more effective against Ps.aeruginosa and B.Subtilis.hence from the experimental data, it may be concluded that furanyl group along with Co^{2+}ion and hence antimicrobial activity it was also observed that 4-dimethylaminobenzionsemicarbazone and O-hydroxybenzoinsemicarbazone and enhance antimicrobial activity when linked to metal ions.

REFERENCES

[1] H.G. pettering and J.A. crime, cancer Res, 27, (1967) 1278

[2] Y. Kumar, S. Chandra; R.P. Singh; A.K. Singh, synthesis and reactivity in Inorganic, Metal - organic and Nano - metal chemistry, 14(2), 185-189, 1984.
[3] M.A. Khan and G.M.Bouet, Transition Met. Chem., 24, 294 - 298, 1999.
[4] B. Kumar, S.K. Sangal; A. Kumar, J. Ind. Chem., Soc., 86, 514 - 517, 2009
[5] V.M. Leovac; L.S. Vojinovic; K.M. Szecsenyi; V.I. cestijevic, J. Serb. Chem. Soc., 68 (12)919-927,2003
[6] Shaikh M. Mohsin Ali, M. Abdul Kalam Azad, MeleYesmin, Shaikh, ShamimAhsan, M. MijanurRahman, YahanAraKhanam, M. Nazrul Islam, Sha M Shahan Shahriar, Asian pacific journal of Tropical Biomedicine, 4, 438 – 442 2012
[7] M. Tyagi and S. Chandra, open J. Inorganic Chem., 2(3), 41 - 48, 2012
a. V.L. Siji, M.R. Sudarsanakumar; S. Suma; A. George; P.V. Thomas, Ind. J. Chem., 50(A), 793 - 797, 2011
[8] J.K. Swearingen and D.X. West, Tranining Met. Chemistry, 26, 252 - 260, 2001
[9] R.K. Agrawal and S. Prasad, Turk J. Chem., 30, 553 - 562, 2006.
[10] A.I. Mikhaleva; A.V. Ivanov; A.M. Vasiltsov. I.A. Vshakov; J.S. Ma; G. Yan, Chemistry of Heterocyclic compounds, 44(9); 1117, 2008
[11] S. Chandra and M. Tyagi, J. Ind. Chem, Soc., 85, 42 - 47, 2008
[12] A.A. El- Asmy; M.E. Khalifa; M.M. Hassanian, Ind. J. of Chem; 43(A), 92 - 97,2004.
[13] M.A. Monshi, J. Ind. Chem. Soc., 75, 158 - 159, 1998.
[14] K. Nakamoto, “Infrared and Raman spectra of Inorganic and coordination compounds” willeyInterscience, New York, 3rd Ed. 171 1978
[15] S. Chandra, L.K. Gupta; K.K. Sharma, J. Saudi Chem. Soc., 9(1), 131 - 136,2005.
[16] P.S. Deshmukh. A.R. Yaul; J.N. Bhojane, A.S. Aswar, world J. Chem, 5(1), 57 and 59.2010
[18]H.M.A. - Fattah; A.L. El-Ansary; N.S. Abdel-Kadu J. Therm Anal. Cal., 96,961, 2009.