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Synthesis and Characterization of Transition Metal Complexes of Substituted Furoinhydrazones

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ABSTRACT

Recently, the synthesis of 2-hydroxybenzoinhydrazone, 2-hydroxybenzoinphenylhydrazone and 2-hydroxybenzoinsemicarbazone with hydrazine hydrate, phenyl hydrazine and semicarbazide hydrochloride in presence of aqueous sodium hydroxide in DMF-water (80%) medium respectively. Similarly, furoinhydrazone, furoinphenylhydrazone, furoinsemicarbazone were synthesized by the interactions of furoinbenzoin with hydrazine hydrate, phenyl hydrazine and semicarbazide hydrochloride in presence of aqueous sodium hydroxide in DMF-water (80%) medium respectively. The synthesis of 2-hydroxybenzoin and furoinbenzoin were carried out by the known literature method. The structure of all the synthesized compounds were justified on the basis of chemical characteristics, elemental and I.R. and NMR spectral analysis.Benzoin hydrazone and its metal complexes were synthesized from substituted benzoinhydrazone. They were characterized by elemental analysis and spectral analysis. The synthesized complexes were screened for antimicrobial activity. At a concentration of 1000µgm/ml. Which was serially diluted to determine their MIC value.

Introduction

The complex formation of benzoinhydrazone with copper(II) and nickel(II) , were synthesized by scherbakov[1] .Prasad[2] , studied by the synthesis of Novel(II) complexes with now ligand derived from hydrazone of isoniazid and their magnetic-spectral, electrochemical, thermal and antimicrobial investigation. Benzoinhydrazone are well known for their biological activity coordination compounds containing ONS as donor atoms are reported to antimicrobial activity[3]. Synthesis, spectral and biological studies of Co(II), Ni(II), Zn(II), Cu(II) and Cd(II) complex with benzyl salicyladehyde acyldihydrazone were carried out by singh[4]. The simultaneous spectroscopic determination of palladium and osmium with salicyladehyde hydrazone was carried out by Ray[5]. The synthesis and structural characterization of three new co-ordination complexes of Co(II), Mn(II) and Cu(II) with N,N,O- donor hydrazine ligands were carried out by shit[6] .The coordination chemistry of hydrazones is an intensive area of study and numerous transition metal complexes of these ligands have been investigated[7]. Synthesis and characterization of some copper(II) complexes with N,S,O-donor thiohydrazones were carried out by Dey[8]. The important reactions of carbonyl with hydroxylamine, semicarbazide and various hydrazines were briefly studied in presence of strong base in ethanol medium.[9-14].While furoinbenzoinoximes, furoinbenzoin hydrazone, furoinphenyl hydrazone and furoinbenzoin semicarbazone were synthesized by the interaction of furoinbenzoin with hydroxylamine hydrochloride, hydrazine hydrate, phenyl hydrazine and semicarbazide hydrochloride in presence of aqueous sodium hydroxide in DMF-Dioxane-water (80%) medium respectively.

Experimental

The melting point of all synthesized compounds was recorded using hot paraffin bath. The carbon and hydrogen analysis were carried out of carlo-Ebra 1106 elemental analyser. Nitrogen estimation was carried out colman-N-analyzer-29. IR spectra were recorded on perkin Elmer spectra were recorded on Brucker Ac 300F spectrometer with TMS as internal standard using $CDCl_3$ and DMSO-d_6 as solvent. The purity of compounds was checked on silica Gel-G pellets by TLC with layer thickness of 0.3mm. All chemicals used were of AR grade. The 4-DMABH was prepared by refluxing substituted benzoine with hydrazine hydrate in presence of alkaline medium for 3-4 hours this reaction mixture was kept overnight. This 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.Synthesis of complxes the equimolar mixture of each of the ligand(0.01M) and metal salts(0.01M) were refluxed on a water bath for 6,8 hours in presence of sodium acetate in ethanol . The reaction mixtures was kept overnight. The product formed were isolated washed several times with cold water ethanol mixture. The characterization of synthesized complex was made with elemental analysis and IR.

IR spectral of ligand 2-HBH show band at 3412cm^{-1} υ(O-H). In complexes 2-2HBH-Co(II) complexes Show band at decreases to 3353cm^{-1} indicating through hydrogen oxygen. However 1607.5cm^{-1} υ(C=N) significantly decreases to 1570.8 cm^{-1} showing linkage through imino nitrogen.

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

Complexes	Colour	Molecular wt	Decomposition temp.°C	Elemental analysis Found/(calculated)%			
				C	H	N	M
2-HBH	Brown	242	80	68.27 (69.42)	7.44 (8.26)	11.57 (11.59)	-
2-HBH-CO(II)	Brown	540.93	284	61.35 (62.11)	3.91 (4.80)	10.34 (10.35)	9.97 (10.89)
2-HBH-Mn(II)	Black	536.93	327	61.74 (62.57)	3.97 (4.84)	10.42 (10.42)	9.31 (10.31)
2-HBH-Fe(III)	Dark Brown	537.84	337	61.63 (62.47)	3.93 (5.83)	10.41 (10.41)	9.46 (10.38)
2-HBH-Cr(III)	Brown	533.99	289	63.01 (62.92)	3.95 (4.86)	10.48 (10.48)	8.85 (9.73)
FUROH	Brown	206	170	57.46 (58.25)	3.90 (4.85)	12.65 (13.59)	-
FUROH-Co(II)	Red	392.93	293	60.14 (61.07)	3.69 (4.58)	6.20 (7.12)	13.01 (14.99)
FUROH-Mn(II)	Grey	388.93	318	60.81 (61.70)	3.73 (4.62)	6.26 (7.19)	13.27 (14.12)
FUROH-Fe(III)	Brown	389.84	334	60.73 (61.56)	3.70 (4.61)	6.24 (7.18)	13.42 (14.32)
FUROH-Cr(III)	Dark brown	385.99	295	61.25 (62.17)	3.81 (4.66)	6.33 (7.25)	12.54 (13.46)

IR spectral data of ligand and its complexes as shown in table-2

Ligands and and its Complexes	υ(o-H)	υ(c=N)	υ(C-O)	υ(M-O)	υ(M-N)
2-HBH	3412	1607	1417.9	-	-
2-HBH-Co(II)	3353	1570.8	1400	507.3	587.1
2-HBH-Mn(II)	3354.1	1568.8	1378.1	504.2	586.8
2-HBH-Fe(III)	3341.0	1558.4	1369.1	476.0	584.0
2-HBH-Cr(III)	3327.4	1555.4	1367.6	475.0	593.4
FUROH	3420	1620.5	1419.0	-	-
FUROH-Co(II)	3284.7	1597.1	1384.2	506.0	586.5
FUROH-Mn(II)	3396.8	1587.2	1369.4	503.7	588.9
FUROH-Fe(III)	3391.4	1581.6	1336.8	485.0	587.0
FUROH-Cr(III)	3393.2	1540.5	1327.3	481.6	585.1

Result and discussion

IR spectra of ligand FUROH show band at 3420 cm^{-1} υ (O-H) in complexes FUROH-Co (II) which decreases to 3284.7cm^{-1} indicating through hydrogen oxygen however 1620.5cm-1 υ(C=N) significantly decreases to 1597.1cm^{-1} showing linkage through imino nitrogen .

In the ligand 2-HBH -&-FUROH and its complex shows a broad band in the region 3515-3863 cm^{-1} may be assigned to υ (O-H) vibration of water molecule. Another sharp and strong bands are observed at 1417-1336 cm^{-1} .similarly the frequencies υ(O-H) υ(C=N) υ(C-O) stretching continuously decresaes to the complex as compaired to ligands band values such as 2-HBH and FUROH and the stretching υ(M-N) and υ(M-O) pattern also decreases in continueous manner in the complexes.

Magnetic moment and electronic spectral data (cm^{-1}) of the metal complexes as given table-2

Complexes	μeff(BM)	λmax(cm^{-1})
2-HBH-Co(II)	4.80	13605,18181,22222
2-HBH-Mn(II)	5.96	13971,118518,22354
2-HBH-Fe(III)	5.65	13513,19607,21978
2-HBH-Cr(III)	4.23	13886,18867,22222
FUROH-Co(II)	2.54	13670,19590,22892
FUROH-Mn(II)	5.89	13870,19194,21739
FUROH-Fe(III)	5.65	13698,18518,22222
FUROH-Cr(III)	4.16	13670,19590,22892

The electronic spectrum of 2-HBH-Co(II) complex exhibits three transition in the range 13605, 18181, 22222cm^{-1}. These spectral bands may be assigned to the following transitions 4_{T1g} (F) $\rightarrow$ 4_{T2g} (F) ,4_{T1g} (F) $\rightarrow 4_{A2g}$ (F), 4_{T1g} (F) $\rightarrow$ 4_{T1g}(p) characteristic to an octahedral geometry[15]

The magnetic moment of 4.80 BM for Co(II) complexes is consistent with octahedral geometry around central metal ion[16] . FUROH-CO (II) complexe exhibit absorption bands at 13670 ,19493 ,22354 $cm^{-1.}$ which may be consign to $4_{A2g}) \rightarrow 4_{T1g}$, $4_{A2}g \rightarrow 4_{T1g}$ (F) & $4_{A2g} \rightarrow 4_{T1g}$ (F) transition respectively [17-18.] suggesting on octahedral geometry on around cobalt (II) ion in the complexes under study furthermore, the magnetic moment measurement recorded at room temp lies at 2.54 BM .This values is indicates of an octahedral geometry of these complex [19-20].

Antimicrobial activity

The compounds were assayed for their antimicrobial activities[21]. Against for test organisms .E.coli,S.aureus, P-aeruginosa and B.subtilis, at a concentration of ***1000μgm/ml by agar well technique***[22]. Further their MIC value against these organisms were determined by serial dilution method using DMF as a solvent, the results obtained are given in the following table.

MIC values in ***μgm/ml*** of compounds

Complexes	E.coli	S.aureus	P. aeruginosa	B.Subtilis
2-HBH-Co(II)	125	63	63	125
2-HBH-Mn(II)	125	250	125	250
2-HBH-Fe(III)	125	63	125	125
2-HBH-Cr(III)	125	125	125	250
FUROH-Co(II)	125	63	125	125
FUROH-Mn(II)	125	125	125	63
FUROH-Fe(III)	125	125	63	63
FUROH-Cr(III)	125	125	125	250

On the basis of MIC values, 2-HBH-Co(II) & FUROH-Fe(III) is found to be most effective antimicrobial agent followed by 2-HBH-Fe (III) & FUROH-Co(II). The enhance antimicrobial activity in care of the compounds 2-HBH-Co(II) may be attributed to the presence of hydroxyl group.

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