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Research Paper

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Synthesis And Studies On Metal Complexes Of 5-(Furan-2-Yl)-3- (2-Hydroxy Phenyl)1-H Pyrazole-1-Carbothiohydrazide

Chemistry

KEYWORDS :chalcone, hiocarbohydrazide, pyrazole -1- carbo thiohydrazide, metal complexes, biological activities

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ABSTRACT

Transition metal complexes of novel pyrazole based heterocyclic ligand 5 - (furan 2-yl) -3- (2 hydroxy phenyl) 1H pyrazole -1- carbo thiohydrazide (ML.H2O); M = Cu(II), Ni (II), Co(II) and Zn(II)) have been synthesized and studied. All the metal complexes have been characterized with the aid of elemental analyses, molar conductance, magnetic susceptibility measurements, IR, UV-Vis.and EPR, mass spectra and cyclic voltammetric studies. The ligand behaves as a tridentate with O, N and S chromophores. The synthesized complexes are found to be monomeric and non-electrolytic nature by magnetic susceptibility and molar conductance data. The UV-Vis., EPR spectral data of the complexes suggest square planar geometry around the central metal ion. The cyclic voltammetric profiles of the complexes show a quasi-reversible peak. The in vitro biological activities of the compounds tested against bacteria such as Gram positive Staphylococcus aureus and Gram negative Escherichia coli and Pseudomonas aeruginosa and antifungal activity against Candida albicans by well diffusion method show that all the compounds possess more biological activity than the free ligand.

1. Introduction

Heterocyclic are abundant in nature and are of great significance to life because their structural sub units exist in many natural products such as vitamins, hormones, antibiotics etc. [1]. Hence, they have attracted considerable attention in the design of biologically active molecules [2]. The chemistry of chalcones has generated intensive scientific studies throughout the world. Especially interest has been focused on the synthesis and biodynamic activities of chalcones. Chalcones are used to synthesize several derivatives like cyanopyridines, pyrazolines isoxazoles, pyrimidines, having different heterocyclic ring systems [3-6]. This report is probably the first example of pyrazoline formation by the reaction of a α, β-unsaturated carbonyl compound with a hydrazine derivative. Synthesis of pyrazolines has been also stimulated by the fact that some of their derivatives were found to possess important bioactivities. Especially their antimicrobial [7], immunosuppressive [8] and central nervous system activity [9] should be emphasized. Although pyrazolines are useful substances in drug research and are well-known five membered nitrogen-containing heterocyclic compounds. In this paper we report the synthesis and characterization of 5-(furan 2yl)-3-(2 hydroxy phenyl)-1H pyrazole - 1 - carbothiohydrazide , and its metal complexes with Co(II), Ni(II) , Cu(II) and Zn(II).

2. Experimental

All chemicals were obtained from Aldrich Chemical & Co. and used without purification. The UV-Vis spectra of the ligand and metal complexes were recorded in DMF using a JASCO V-530 spectrophotometer. IR spectra in KBr discs were recorded on a JASCO FT-IR 460 plus spectrophotometer at Thiagarajar College, Madurai. Elemental analyses were performed at SAIF, CDRI, Lucknow. 1H-NMR spectrum of the ligand were recorded in $CDCl_3$ using a Bruker DRX-300, 300MHz NMR spectrometer. EI mass spectra were recorded at IIT, Madras. Cyclic voltammetry measurements were carried out at room temperature in DMS (CH Instruments, USA, voltammograph) using a three-electrode cell containing a reference Ag/AgCl electrode, Pt wire auxiliary electrode, and glassy carbon working electrode with tetrabutylammonium perchlorate (TBAP) as supporting electrolyte. EPR spectrum was recorded at SAIF, IIT, Bombay. Magnetic moments of the complexes were measured on a Magnetic Susceptibility Balance Mark 1 Sherwood UK at Thiagarajar College, Madurai. Effective magnetic moments were calculated using the formula $\mu_{eff} = 2.828 (\chi_M T)^{1/2}$ where χ_M is the molar susceptibility. Molar conductance of the complexes (10^{-3} $molL^{-1}$) was measured in DMF at room temperature using a Systronic conductivity bridge.

2.1 Synthesis of ligand 5 - (furan 2yl) -3 (2 hydroxy phenyl)- 1H pyrazole - 1 - carbo thiohydrazide[FHPCT]

The synthesis of chalcone (E)-3-(furan-2-yl)-1-(2-hydroxyphenyl)prop-2-en-1-one from furfuraldehyde and 2- hydroxy acetophenone by the Claisen- Schmidt conden sation method [10-12].

To a suspension of chalcone (10mmol) and concentrated hydrochloric acid (0.5ml) in 25ml of ethanol, thiocarbohydrazide (10mmol) was added. The mixture was refluxed for 10h. The products were poured into crushed ice and the solid separated out was filtered dried and recrystallized from ethanol. The Schematic representation of the ligand is given in the figure 1. Yield : 93%, Melting point : 700C

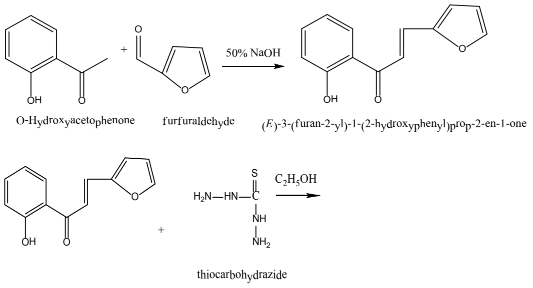
2.2. Synthesis of complexes

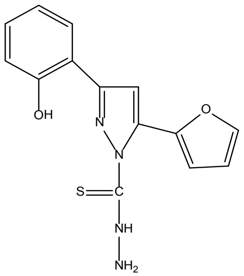
The solution of $CuCl_2$. $2H_2O$(10 mmol) in 10mL of ethanol was added to 30mL solution of (10 mmol) of FHPCT in ethanol. The mixture was stirred for 3 h and the green precipitate was obtained and filtered, washed with water and dried with ethanol. Other complexes of $CoCl_2.6H_2O$, $ZnCl_2$, were prepared by the same procedure.

The nickel(II) complex was prepared by10 mmol of $NiCl_2$. $6H_2O$ in 15mL of hot ethanol and refluxed with 10 mmol of FHPCT at 50–600C for 2 h. The precipitated of green nickel complex was filtered and washed with water and then dried with ethanol.

3. Results and discussion

Ligand FHPCT is prepared via condensation of o-hydroxyacetophenone and furfuraldehyde followed by the substitution reaction with thiocarbohydrazide. The metal complexes $ML.H_2O$ are prepared, and characterized by using conductometry, elemental analysis and spectral studies. The analytical data and physical properties of FHPCT and its metal complexes are presented in Table 1.





5-(furan-2-yl)-3-(2-hydroxyphenyl)-1*H*-pyrazole-1-carbothiohydrazide

[FHPCT]

Figure 1 : The Schematic representation of the ligand [FHPCT] Elemental analyses suggests that the complexes have 1 : 1 (metal : ligand) stoichiometry. The analytical data of the complexes are in good agreement with the general formula [ML. H2O], where M= Cu(II), Ni(II), Co(II) and Zn(II). The magnetic moments show paramagnetic Cu(II), Co(II), and Ni(II) and are in agreement with four-coordinate square planer metal (figure 2) geometry.

Table.1.Elemental analysis, Magnetic susceptibility, molar conductance of the ligand and metal complexes

Compound /complex	Formula weight	Color	m.p /°C	Analysis found (Calculated) (%)							
				M	C	H	N	O	S	µeff. (B.M)	Molar conductance Λm Ohm/ cm2 mol-1
C14H12N4O2S	300.07	Yellowish brown	700C	-	55.81 (55.99)	4.00 (4.03)	18.60 (18.65)	10.58 (10.65)	10.61 (10.68)	-	-
CuC14H14N4O3S	381.90	Green	>3600C	16.55 (16.68)	44.00 (44.15)	3.62 (3.44)	14.65 (14.71)	12.56 (12.60)	8.34 (8.42)	1.74	18
CoC14H14N4O3S	377.28	Reddish brown	>3600C	15.48 (15.66)	44.42 (44.69)	3.40 (3.48)	14.82 (14.85)	14.69 (12.76)	8.44 (8.52)	1.87	21
NiC14H14N4O3S	377.04	Dark green	>3600C	15.50 (15.61)	44.57 (44.72)	3.65 (3.48)	14.82 (14.90)	12.70 (12.76)	8.46 (8.53)	0	19
ZnC14H14N4O3S	383.76	Greenish Yellow	>3600C	16.98 (17.09)	43.78 (43.93)	3.58 (3.42)	14.58 (14.64)	12.48 (12.54)	8.30 (8.38)	Diamagnetic	17

3.1 Electronic spectra

The electronic absorption spectra of the Ligand [FHPCT] and its Cu(II), Co(II) and Ni(II) complexes were recorded at room temperature using DMF as the solvent. The electronic spectral data of that ligand and metal complexes are given Table.2

The electronic spectrum of the free ligand shows a bands at 43592 cm–1, 28490 cm–1 which is assigned as intra-ligand charge transfer band (INCT). In the electronic spectrum of the Cu(II) complex was noticed the presence of a new bands at 14579cm–1 and 22779 cm–1, assigned to the 2B1g → 2B2g ,2B1g → 2Eg. transition . These transitions, as well as the measured value of the magnetic moment (1.74 B.M) suggest a square-planar.

In the electronic spectrum of Co(II) complex, two new absorption bands are observed at 16286 cm–1 and 27900 cm–1. These bands are assigned to 1A1g → 1B1g . and INCT transitions, respectively. These transitions indicate a square-planar geometry of the complexes. The observed magnetic moment, 1.87 B.M is in agreement with this structure. The electronic spectrum of Ni(II) complex shows two new bands at 14084cm–1 and 12195cm–1,which are attributed to 1A1g → 1B1g and 1A1g → 1B2g transitions, respectively. These transitions and the magnetic moments (µeff = 0) suggest a square-planar stereochemistry. Finally, the magnetic moments of the Zn(II) complexe were found to be diamagnetic, while all other complexes were paramagnetic with magnetic moment values close to the spin only values. Zn(II) are suggested to possess a square-planar environment in the complex. [10-12]

Table 2: Electronic spectral data of the ligand and its metal complexes

S.No.	Compound	Frequency(cm^{-1})	Assignment	**Geometry**
1	[FHPCT]	43592 28490	INCT INCT	-
2	[CuFHPCT. H_2O]	14579 22779	$^2B_{1g} \rightarrow {}^2B_{2g}$ $^2B_{1g} \rightarrow {}^2E_g$	Square Planar
3	[CoFHPCT. H_2O]	16286 27900	$^1A_{1g} \rightarrow {}^1B_{1g}$ INCT	Square Planar
4	[NiFHPCT. H_2O]	18800 21650	$^1A_{1g} \rightarrow {}^1B_{1g}$ $^1A_{1g} \rightarrow {}^1B_{2g}$	Square Planar

3.2. IR spectra

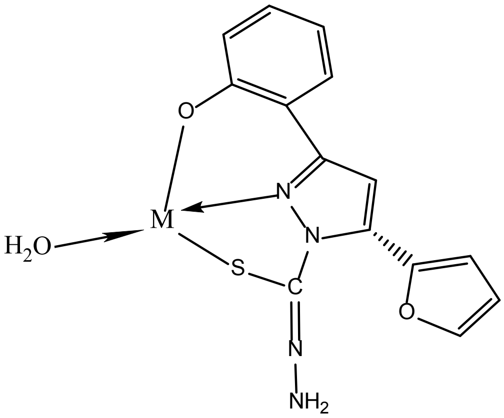
The important IR frequencies exhibited by the ligand [FHPCT] and its complexes are given in the Table 3. The ligand exhibit tautomerism, and one can expect both S-H band and C=S band [13]. A medium intensity band around 1533 cm-1 shows C-S-H linkage which shows at 1531 cm-1 in parent compound thiocarbohydrazide. Also the weak band at 1262 cm-1 ascribed –C-S-N linkage, which is a shown at similar frequency in parent compound thiocarbohydrazide. A weak band at 2198 cm-1 may be ascribed to the single bond C-S vibration due to its thione-thiol tautomerism form of the ligand.

The Infrared spectrum of ligand exhibits high intensity band around 1595 cm-1 is due C=N stretching frequency. The C=N (ring) stretching frequency in the ligand shifts to 1626 cm-1 in the complexes, showing coordination of pyrazole nitrogen with the metal ion.[14]. In addition, IR spectrum of the ligand revealed a sharp band at 1294 cm-1 due to C=S of side chain, which is shifted to lower frequency about 20-40 cm-1. In all complexes, these indicate sulphur atom of the side chain also involved bond formation with metal ion. The phenolic OH band at 3219 cm-1 in the ligand, disappeared in complexes, which indicates the ligand coordinate to the metal ion through phenolic oxygen atom via deprotonation [15]. A broad band appeared in the region 3416-3443 cm-1 in all complexes indicates the presence of coordinated water molecule [16,17]. The new band observed in region at 756-748 cm-1, 594 -596 cm-1 and 470 - 470 cm-1can be attributed to M-O, M-N and M-S bonds.

S. No	Compound	Frequency(cm-1)						
		ν(O-H)	ν (C=N)	ν(C-N)	ν (C=S)	ν(M-O)	ν(M-N)	ν(M-S)
1	[FHPCT]	3219	1595	1469	1294	-	-	-
2	[CuFHPCT. H2O]	3443	1606	1452	1232	756	594	430

3	[CoFHPCT. H2O]	3416	1626	1489	1253	752	594	470
4	[NiFHPCT. H2O]	3419	1607	1471	1151	758	594	468
5	[ZnFHPCT. H2O]	3420	1626	1452	1240	756	596	460

Table 3 : The IR spectrum (cm-1) of the lignd and metal(II) complexes



M = Cu(II), Co(II), Ni(II), Zn(II),

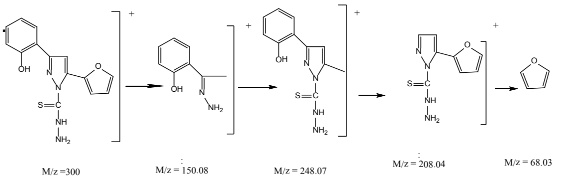
Figure 2. Proposed structure of M (II) complex of [FHPCT]

3.3. 1H-NMR spectroscopy

The 1H-NMR spectrum of the ligand was recorded in CDCl3 solution. The spectrum of the free ligand, showed the multiplets observed at 6.53 ppm and 7.36 ppm due to aromatic protons and the –OH group resonance signal is appeared as a singlet at 10.95 ppm. Also another one singlet is appeared due to the SH at 10.15 ppm. The doublet at 3.70 and 3.72 ppm due to the –CH=CH proton and the triplet at 2.26 , 2.32 and 2.39 ppm due to CH2 proton.

3.4. Mass spectra

Mass spectra provide a vital clue for elucidating the structure of compounds. The mass spectra of the ligand and its zinc complex were recorded and used to compare their stoichiometry composition. The ligand showed a molecular ion peak at m/z 300 corresponding to [C14H12N4O2S]+ ion. Also, the spectrum exhibited the fragments at m/z 248, 207, 150, corresponding to [C11H12N4OS]+, [C8H8N4OS]+, and [C8H10N2O]+ respectively. The mass spectrum of Zn(II) complex showed peak at m/z 383 which may represent the molecular ion peak. The Zn(II) complex underwent demetallation to form the species [L]+, gave fragment ion peak at m/z 383 which is corresponding to the original molecular weight of the Ligand under investigation. Which corresponds to mass of Zn2+ complex. The other fragments were observed at m/z 219, 154, 127 and 81



3.5. EPR spectra

EPR spectra of copper complexes provide important information for studying the metal ion environment. The X-band EPR spectrum of Cu(II) complex was recorded in DMSO at liquid nitrogen temperature and room temperature (300 K). The spin Hamiltonian parameters calculated for the copper complex are given in Table 4. The spectrum of the copper complex at room temperature shows one intense absorption in the high field and is isotropic due to tumbling of the molecules. However, this complex in frozen state shows four well-resolved peaks at low field. In square planar complexes, the unpaired electron lies in the d x2-y2 orbital giving g∥ (2.303) > g⊥ (2.058) > ge(2.0023). The fact g∥ > g⊥ suggests that the complex is square planar. This is also supported by the fact that the unpaired electron lies predominately in the d x2-y2 that is evident from the value of the exchange interaction term G, If G >4.0, the local axes are aligned parallel or only slightly misaligned. If G <4.0, significant exchange coupling is present and the misalignment is appreciable. The observed value for the exchange interaction parameter for the copper complex (G = 5.398) suggest that the local tetragonal axes are aligned parallel or slightly misaligned and the unpaired electron is in d x2-y2 orbital [18–21]. The spin orbit coupling constant, λ value (– 400 nm) calculated using the relations, gav =1/3(g∥)+2/3(g⊥), is less than the free Cu(II) ion (–832 nm) which also supports the covalent character of M–L bond in the complex. The covalence parameter α2 is calculated using the following equation:

$$\alpha 2Cu = (A|| / p) + (g|| - 2.0023) + 3/7(g\perp - 2.0023) + 0.04$$

If the α2 =0.5, it indicates complete covalent bonding, while the value of α2 =1 suggests complete ionic bonding. The observed value 0.861 of α2 of the complex indicates that the complex has some covalent character in the ligand environment [22] .

Table 4. EPR spectral data of copper(II) complex

Compound	$g_{\parallel}$	$g_{\perp}$	g_{av}	α^2	$A_{\parallel}$ x10^{-4} cm^{-1}	$A_{\perp}$ x10^{-4} cm^{-1}	$g_{\parallel}$ /$A_{\perp}$
	2.303	2.058	2.135	0.861	178.920	35.725	136.336

3.6. Electrochemical studies

3.6.1. Cyclic voltammetry

The free ligand does not show oxidation or reduction peak values but controlled potential electrolysis indicates that each process corresponds to one electron transfer processes. The cyclic voltammograms of copper(II) complex (10-3 M) in DMSO scan rate 100 mVs-1 shows a well defined redox process corresponding to the formation of Cu(II)/Cu(I) couple at Epa = 0.454 V and Epc =0.583 V versus Ag/AgCl which are similar to the value reported earlier[23]. Copper complex have large separation between cathodic and anodic peak (ΔEp = 0.129 V) indicating the quasi-reversible character [24].

3.7. Molar conductance

The molar conductance value (17 to 21 Ohm-1cm2mol-1) of the complexes which was carried out in DMSO solvent at tempetrure.The molar conductance value indicates indicate that the complex under study is non electrolytic nature. The obtained value suggests that no anions present outside the coordination sphere.

3.8. Biological activities

Antimicrobial activities of the FHPCT and its metal(II) complexes were tested in vitro by the well diffusion method [25] against gram positive bacteria like Staphylococcus aureus, gram negative bacteria like Pseudomonas aeruginosa, Escherichia coli and antifungal activity against Candida albicans. The test solutions were prepared in DMSO, nutrient agar used as culture medium. The zone of inhibition was measured in mm and the values of the investigated compounds are summarized in Table 5. From the observed result it is clear that the all the metal(II) complexes showed enhanced antimicrobial activity than that of free ligand (FHPCT). The Co(II) complex has some significant activity. Such an increased activity of the complexes can be explained on the basis of Overtone's concept [26] and Tweedy's chelation theory [27]. A possible explanation for the observed increased activity upon chelation is that the positive charge of the metal in chelated complex is partially shared with the ligands donor atoms so that there is an electron delocalization over the whole chelate ring. This, in turn, will increase the lipophilic character of the metal chelate and favors its permeation through the lipoid layers of the bacterial membranes.

Table 5 Antimicrobial activity of the ligands and metal complexes (zone of inhibition in mm).

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Compound	Zone of inhibition (in mm)			
	Gram(+)	Gram(-)		Fungi
	Staphylococcus aureus	Escherichia coli	Pseudomonas auringosa	Candida albicans
Ligand	14	R	12	R
[CuFHPCT. H2O]	16	14	16	17
[CoFHPCT. H2O]	14	14	18	20
[NiFHPCT. H2O]	17	16	17	16
[ZnFHPCT. H2O]	18	12	16	18
Amikacin	20	25	22	-
Ketokonazole	-	-	-	25

4.0 CONCLUSION:

Complexes of 5 - (furan 2yl) -3 (2 hydroxy phenyl) 1H pyrazole - 1 - carbo thiohydrazide with Cu(II), Co(II),Ni(II) and Zn(II), were synthesized and characterized by various physical–chemical methods. The ligand coordinating through pyrazole ring nitrogen and side chain of the sulphur and oxygen. From the spectral data, all the metal(II) complexes of ligand [FHPCT] exhibit square planar geometrical arrangements. In addition, the antimicrobial activity study indicates that the complexes showed more activity than ligand has less activity against some microorganisms under identical experimental conditions.

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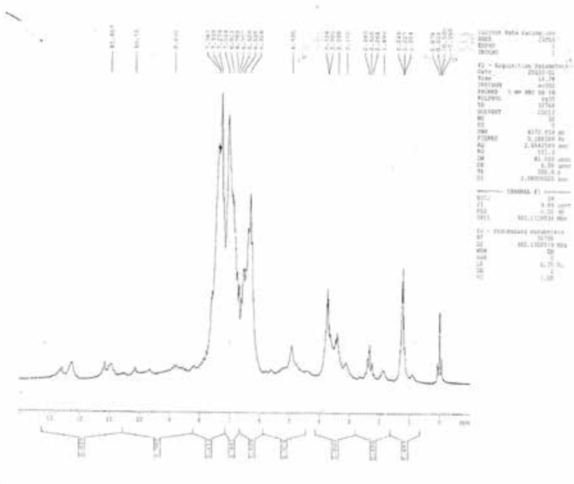

Figure 3. The 1H NMR Spectrum of the ligand [FHPCT]

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