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Spectral studies, synthesis and charaterization of Co (II), Ni (II) and Cu (II) complexes with biologically active Schiff base ligands

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Abstract

The complexes of Co (II), Ni (II) and Cu (II) having general formula [M (PSC)$_2$] and [M (PTC)$_2$], where M = Co (II), Ni (II), Cu (II) and PSC=α-pyridone semicarbazone, PTC=α-pyridone thiosemicarbazone been synthesized. The ligands as well as complexes have been characterized by elemental analyses, molar conductance, magnetic susceptibility, infrared spectra and electronic spectral data, PSC and PTC acts as a tridentate ligand and coordination proposes through N of pyridone ring, azomethine nitrogen and carbonyl oxygen/ thoine sulphur of semicarbazone/thiosemicarbazone moiety. On the basis of electronic spectral and magnetic susceptibility values the geometry of the complexes were proposed to be octahedral. The molar conductivity values of the complexes were found to be non-electrolytic in nature. The ligand and complexes have been screened for their antibacterial and antifungal activities.

Keywords: PSC, PTC, schiff base, complexes, antimicrobial study

Introduction

Pyridine derivative is of great importance for the medicinal chemists because of their diverse type of pharmacological activities. They play vital role in biological field such asantiallergic[1], antibacetrial[2], fungicidal[3], antitubercular[4], antiviral[5], antiinflammatory[6], anticancer[7] and anthelmintic[8] activities. In the present manuscript mainly deals with the spectral studies, synthesis and characterization of Co (II), Ni (II) and Cu (II) complexes with biologically active Shiff base ligands, α-pyridone semicarbazone and α-pyridone thiosemi carbazone are reported here.

Materials and Methods

All the used chemicals were of AR grade BDH reagents. The complexes were analysed using standard procedures [9], IR spectra were recorded on a Perkin Elmer-577 IR spectrophotometer in KBr medium. The conductivity measurements made on a systronics conductometer model 303 using DMF as the solvent. Magnetic susceptibility was measured by Gouy method using mercury tetraisothiocynatocobaltate as the calibrant. The electronic spectra were recorded on Cary-2390 spectrophotometer. Analytical data, colour, mol. wt., magnetic susceptibility, decomposition temperature and electronic spectral data are recorded in Table-1 and salient features of IR spectral data are recorded in Table-2.

Preparation of the Ligands

The Schiff bases wereprepared in 75-80% yield by condensation of ethanolic solution of α-pyridone (0.01M) with semicarbazide hydrochloride/ thiose micarbazide. hydrochloride (0.01M) dissolved in minimum amount of sodium acetate in tetrahydrofuran. The resulting mixtures were refluxed on water bath for 3-4 hrs with frequent stirring. On cooling, the precipitate separated, filtered and washed several times with alcohol and dried and recrystallized with tetrahydrofuran. Both the compounds are colourless needles. m.p. -136±1^{0}C for semicarbazone ligand and 148± 1^{0}C thiosemicarbazone ligand.

Preparation of the complexes

The complexes were prepared by reacting respective metal acetates of Co (II), Ni (II) and Cu (II) in ethanolic solution of the ligands of α-pyridone semicarbazone/thiosemicarbazone in molar ratio 1:2. The resulting reaction mixtures were refluxed on water bath for 2-3 hrs. The procedure carried out in each case was similar with slight variation of timing of reflux. After cooling solid complexes separated out which were filtered, washed with ethanol, dried and recrystallised with tetrahydrofuran. Yield in all cases was 70-75%. All the complexes were found to be insoluble in water and also in most of the common organic solvents.

Results and Discussion

The IR spectra of the ligand PSC/PTC show a short broad band at 3400 and 3480 cm^{-1} respectively, assigned [10] to ν_{N-H} After complexation this band shifted to lower frequency region with slight reduced intensity. This proposes linkage with the

N atom of pyridone ring. The next IR band of structural significance in the spectra of the ligand's PSC/PTC appears at ~ 1500cm^{-1}, assigned [11] to $V_{C=N}$ This band also suffered a downward shift in complexes indicating the coordination of the azomethine N atom with either semicarbazone or thiosemicarbazone group.

Table 1: Analytical, color, mol.wt., magnetic susceptibility, electronic spectra conductivity measurements and descomposition temperature of the ligand PSC/PTC and its metal complexes.

S. No.		Mol.wt.	% Analysis found (calculated)				μ_{eff} B.M.	λ_{max} Electronic cm^{-1}	Ω_m Ohm^{-1} cm^{2}mol^{-1}	D.T^{0}C
			M	C	N	H				
1.	PSC (colorless)	152	-	47.48 (47.36)	36.72 (36.84)	5.19 (5.26)	-	-	-	-
2.	PTC (colorless)	168	-	42.96 (42.85)	33.25 (33.33)	4.68 (4.26)	-	-	-	-
3.	[Co (PSC)$_2$] (yellowish red)	360.93	16.23 (16.23)	40.01 (39.89)	30.96 (31.03)	4.38 (4.43)	4.87	12700, 17300	3.3	212
4.	[Co (PTC)$_2$] (Greenish red)	382.93	15.44 (15.44)	37.52 (37.90)	29.16 (29.24)	4.11 (4.17)	4.92	12300, 17400	3.7	215
5.	[Ni (PSC)$_2$] (Reddish brown)	360.71	16.35 (16.27)	39.89 (39.92)	30.96 (31.04)	4.38 (4.43)	3.04	10300, 16200, 22300	4.2	237
6.	[Ni (PTC)$_2$] (Brown)	382.71	15.40 (15.34)	37.73 (37.62)	29.35 (29.26)	3.61 (3.65)	3.01	10100, 15900, 22100	4.9	231
7.	[Cu (PSC)$_2$] (Blue)	365.54	17.26 (17.38)	39.28 (39.24)	30.74 (20.63)	3.94 (3.82)	1.83	11200, 18200	5.3	240
8.	[Cu (PTC)$_2$] (blue)	397.54	16.04 (15.98)	36.14 (36.22)	38.09 (28.17)	3.46 (3.52)	1.90	11400, 18400	5.4	247

D.T. = Decomposition temperature.

Table 2: Salient features of IR spectral bands of ligands PSC and its metal complexes

S.No.	Compounds	ν_{N-H}	$\nu_{C=H}$	$\nu_{c=o}$	$\nu_{c=s}$	ν_{M-O}	ν_{M-S}	ν_{M-N}
1.	PSC	3400 s, b	1520 s, b	1670 s, m	-	-	-	-
2.	PTC	3480 s, b	1500 s, b	-	820 s, b	-	-	-
3.	[Co (PSC)$_2$]	3370 m, b	1495 m, b	1645 m, b	-	530 m	-	410 m
4.	[Co (PTC)$_2$]	3455 m, b	1470 m, b	-	795 m, b	-	450 m	415m
5.	[Ni (PSC)$_2$]	3375 m, b	1490 m, b	1640 m, b	-	515 m	-	395m
6.	[Ni (PTC)$_2$]	3450 m, b	1475 m, b	-	790 m, b	-	470m	400m
7.	[Cu (PSC)$_2$]	3370 m, b	1490 m, b	1640 m, b	-	510 m	-	405m
8.	[Cu (PTC)$_2$]	3450 m, b	1470 m, b	-	795 m, b	-	480m	420m

m= medium, s=strong, b=broad

The spectrum of the ligand PSC also shows a sharp band of medium intensity at 1670 cm^{-1} which can be assigned [12] to $V_{c=o}$ in the spectra of the complexes, this band has shifted to a higher wave number with change in intensity. The shift of the band and change in intensity indicates coordination of > c = 0 group of semicarbazone to the metal ion. The spectrum of the ligand PTC shows a strong and broad band ~820 cm^{-1} assigned [13] to $V_{c=s.}$ *In* the spectra of the complexes this band suffers downward shift suggesting linkage of the metal ion through thione S atom of thiosemicarbazone group.

The conclusive evidence of bonding of ligand to metal ion is indicated by the appearance of bands due to ν_{M-O} [14] at 530-510 cm^{-1} region, ν_{M-N} [15] at 420 - 395cm^{-1} V_{M-S} [16] at 480-450 cm^{-1}. These comparable with other reported complexes.

The electronic spectral and magnetic susceptibility values (Table-1) proposes octahedral geometry of the complexes which is also supported by low value of molar conductance which indicate non-electrolytic nature [17] of the complexes.

Thus, on the basis of above studies, it is concluded that the ligands PSC/PTC acts in tridentate manner and coordination may takes place through azomethine N atom, N atom of pyridone ring and carbonyl oxygen/thione sulphur atom of either semicarbazone/thiosemicarbazone group.

Thus, on the basis of above studies it is concluded that the complexes possess the octahedral geometry as shown in Figure-1.

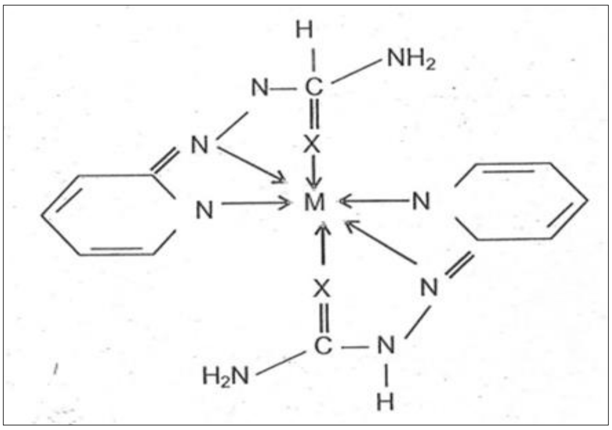

Fig 1: [M(PSC)] and [M(PTC)]; M = Co (II) Ni (II) and Cu (II); x = oxygen or sulphur

Antimicrobial activity: The antimicrobial activity of ligands and their metal chelates were checked against gram negative bacteria *Escherichia coli* and antifungal activity against fungi, *Aspergillus flevus* and *Aspergillus niger*. The antimicrobial and antifungal effect were observed by standard microbiological parameters, and dithane Z-78, using disc diffusion technique [18]. Filter paper discs of diameter 12 mm were used and the diameters of zones inhibition formed around each disc after incubating for 72 h at 25-30^{0}C

It was observed that on comparison with reference to antibiotic and fungicides the complexes were found to be more effective than ligands. It is known that chelation tends to make the ligands act as more potent bacterial agents, than the free ligand. The antimicrobial activity of the compounds increases after chelation. Chelation reduces the polarity of the central metal ion by partial sharing of its positive charge with the donor groups [19] increasing lipophilic nature of the central metal ion, which in turn favours its permeation to the lipid layer of the membrance. Other factors, such as solubility, molar conductivity and magnetic susceptibility are also responsible for increasing the antimicrobial activity of the complexes [20]

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