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Synthesis, Characterization and Thermal Investigation of Copper(II), Nickel(II), Cobalt(II) and Zinc(II) Complexes with 5-BenzoyI-1-(phenyImethylenamino)-4-phenyI-1H-pyrimidi

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Synthesis, Characterization, and Thermal Investigation of Copper(II), Nickel(II), Cobalt(II), and Zinc(II) Complexes with 5-Benzoyl-1-(phenylmethylenamino)-4phenyl-1H-pyrimidine-2-thione

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ABSTRACT

Cu(II), Ni(II), Co(II) and Zn(II) complexes of 5-benzoyl-1-(phenylmethylenamino)-4-phenyl-1H-pyrimidine-2-thione were prepared. These complexes have been characterized by elemental analyses, IR, NMR and UV-Vis spectra. Spectral studies, magnetic moment and molar conductivity measurements were performed on these compounds. All of the complexes are non-electrolytes. The metal complexes are proposed

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to have an octahedral structure. Thermal studies of the complexes are also reported.

Key Words: Pyrimidine-2-thione; Metal complexes.

INTRODUCTION

In recent years, heterocyclic Schiff bases containing NS and NSO donor atoms and their 3d metal complexes have attracted considerable attention due to their remarkable antifungal, antibacterial and antitumor activity.^[1,2] The majority of papers of some authors contain the results of spectroscopic and magnetic studies of coordination compounds of heterocyclic^[3–5] derivatives in the solid state. Transition metal compounds with pyrimidine derivatives included in nucleic acids, such as uracyl, cytosine and thymine were also of special interest.

We recently reported the synthesis and characterization of heterocyclic complexes obtained from N-aminopyrimidine-2-one with salicylaldehyde^[6,7] (Figure 1). In this paper we report the preparation, characterization and thermal investigation of new Cu(II), Ni(II), Co(II) and Zn(II) metal complexes.

EXPERIMENTAL

Materials and Methods

MeOH, EtOH, CHCl₃, DMF, toluene, diethylethere, *n*-heptane, thiosemicarbazide and benzaldehyde were obtained from Fluka or Aldrich Chemical Co. The metal salts $Cu(AcO)_2 \cdot H_2O$, $Co(AcO)_2 \cdot 4H_2O$, Zn- $(AcO)_2 \cdot 2H_2O$ and $NiCl_2 \cdot 6H_2O$ were obtained from E. Merck Chemical Co. All solvents were distilled before use.



Figure 1. Structure of the ligand.

Elemental analyses (C, H, N) were performed by using a Carlo Erba 1106 elemental analyzer. The IR spectra were obtained using KBr discs $(4000-400 \text{ cm}^{-1})$ on a Bio-Rad-Win-IR spectrophotometer. The electronic spectra in the 200–900 nm range were obtained in DMF on a Unicam UV2-100 UV/Visible spectrophotometer. Magnetic measurements were carried out by the Gouy method using Hg[Co(SCN)₄] as calibrant. Molar conductances of the Schiff base ligand and their transition metal complexes were determined in DMF at room temperature using a Jenway model 4070 conductivity meter. The ¹H NMR spectra of the Schiff base was recorded with a Varian XL-200 NMR instrument. TGA measurements were carried out by a Shimadzu-50 thermal analyzer.

Synthesis of the Ligand

The Schiff base ligand (5-benzoyl-1-(phenylmethylenamino)-4-phenyl-1H-pyrimidine-2-thione) was synthesized according to the literature method.^[8] The ligand was prepared by the reaction of the 5-benzoyl-4-phenyl-2,3-furandion (1 mmole, 0.395 g) and benzaldehydethiosemicarbazone (1 mmole, 0.180 g) in dry toluene (40 mL), by boiling the mixture under reflux for 1.5 h. The precipitated ligand was filtered, recrystallized from acetic acid and dried in a vacuum dessicator, mp 185°C. Anal. Found: C, 72.70; H, 4.36; N, 10.65; S, 7.81; calc. for $C_{24}H_{17}N_3OS$ (MW 395.5): C, 72.89; H, 4.33; N, 10.63; S, 8.11 %. Yield; 0.140 g, 36%.

Synthesis of the Complexes

The complexes were prepared by adding a methanolic solution (15 mL) of the metal salt (0.02 mole of copper or zinc salt or 0.01 mole of cobalt or nickel salt, respectively) to the solution of 0.02 mole of the ligand in chloroform (30 mL). The precipitated complexes were filtered, washed with water, *n*-heptan, followed by cold methanol and dried in a vacuum dessicator.

RESULTS AND DISCUSSION

All of the synthesized complexes are colored solids. The compounds are stable at room temperature and in air, soluble in DMSO and DMF, but poorly soluble in methanol and ethanol. The elemental analyses and thermal analyses are in agreement with the chemical formulas of the compounds. Copyright © 2003 by Marcel Dekker, Inc. All rights reserved

The reactions of the Schiff base ligand with the corresponding metal salt can be represented as follows:

 $2L + M(AcO)_2 \cdot nH_2O \xrightarrow{\text{methanol/chloroform}} [M(L)_2(AcO)_2] \cdot mH_2O + xH_2O$ $M = Cu(II), Co(II), Zn(II) \qquad L = C_{24}H_{17}N_3OS$ $n = 1 \quad 4 \quad 2$ $m = 1 \quad - \quad x = - \quad 4 \quad 2$ $2L + \text{NiCl}_2 \cdot 6H_2O \xrightarrow{\text{methanol/chloroform}} [\text{Ni}(L)_2\text{Cl}_2] + 6H_2O$

The metal-to-ligand ratio of the bidentate Co(II), Cu(II), Zn(II) and Ni(II) complexes was found to be 1:2, in addition to the two acetate/ chloride ligands. The Cu(II) complex has one additional water of water of crystallization (Table 1).

IR spectra

The infrared values of the complexes studied are presented in Table 2. The IR spectrum of the ligand shows three strong bands at 1650 v(C = O), 1035 v(C=S) and 1600 cm⁻¹ v(C=N) azomethine vibrations.^[6-8] The strong and broad absorption in the region 3440 cm⁻¹ of the Cu(II) complex substantiates the presence of a water molecule.^[13] The band at 1600 cm⁻¹, due to the stretching mode of the azomethine group in the spectrum of the free ligand, shows a remarkable negative shift with splitting in the 1565 cm⁻¹ region in all of the complexes spectra suggesting that the coordinating azomethine nitrogen atom of the Schiff base is involved in the complexes formation. The band at 1035 cm⁻¹ may be attributed to the predominant v(C=S) stretching mode in most of this ligand. The decrease of ~ 30 cm⁻¹ in the frequency of this band in the complexes suggests the coordination of sulfur of the C=S (thione) moiety. On the other hand, the IR spectra of the Co(II), Cu(II) and Zn(II) complexes confirm that the acetate group which may be assigned to AcO anion at ~1415 cm⁻¹ is coordinated.^[13] New bands observed in the complexes at 445–476 cm⁻¹ and 410–425 cm⁻¹ are tentatively assigned to the v(M–N)^[6,7,13] and v(M–S) vibrations.^[13]

Electronic Spectra

The electronic absorption spectral data for the complexes, that are dissolved in DMF, are shown in Table 2. In the spectrum of the Schiff base,

	Table 1.	Analytic	al data and so	me physica	l properties	of the com	plexes.		
	Vield	ہ =		чM	Ele	emental ana (calculat	ulyses foun ted) %	q	Ŷ
Compounds	%	B.M]	Color	°C)	С	Η	z	S	$(S \text{ cm}^2 \text{ mol}^{-1})$
$[Cu(L)_2(AcO)_2] \cdot H_2O$	75	1.72	Light	220	63.5	3.9	8.8	6.7	2.8
C ₅₂ H ₄₂ CuN ₆ O ₇ S ₂ (989.54 g/mole)			brown		(63.0)	(4.2)	(8.4)	(6.4)	
$[Co(L)_2(AcO)_2]$	73	4.89	Dark	244	65.1	3.9	8.9	6.8	3.0
$C_{52}H_{40}CoN_6O_6S_2$ (966.93 g/mole)			green		(64.5)	(4.1)	(8.6)	(9.9)	
$[Ni(L)_2Cl_2]$	65	5.96	Milk-	213	63.0	3.6	9.4	7.3	12.3
$C_{48}H_{34}Cl_3N_6NiO_2S_2$ (919.71 g/mole)			brown		(62.6)	(3.6)	(9.1)	(6.9)	
$[Zn(L)_2(AcO)_2]$	82	Dia	Yellow	237	64.7	3.8	9.0	6.8	2.1
C ₅₂ H ₄₀ N ₆ O ₆ S ₂ Zn (973.37 g/mole)					(64.1)	(4.1)	(8.6)	(6.5)	

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Table 2.	Characteristic IR	and electronic al	bsorption spectral d	lata of the liga	nd and their m	etal complexes.
Compounds	C=S	C=N	OH/H ₂ O	M-N	M-S	$\begin{array}{l} \lambda_{max} \ (nm) \\ (\epsilon^{in} \ (M^{-1} cm^{-1}) \end{array} \end{array}$
$[Cu(L)_2(AcO)_2] \cdot H_2O$	1007 s	1565 m	3440 m,br	455 w	420 w	660 (sh), 593 (34), 480 (339), 325 (1557)
$[Co(L)_2(AcO)_2]$	1008 s	1565 m	I	450 w	410 w	763 (3), 594 (35), 377 (2013)
$[Ni(L)_2Cl_2]$	1002 m	1564 s	I	476 w	413 w	660 (sh), 542 (18), 405 (902) 325 (2865)
$[Zn(L)_2(AcO)_2]$	1007 s	1565 m	I	445 w	425 w	395 (1280), 322 (2230)

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the bands of 300–400 nm are attributed to the azomethine chromophor and benzenoid $\pi \to \pi^*$ and $n \to \pi^*$ transitions.

The six coordinated Cu(II) complex is expected to be tetragonal and is confirmed by the characteristic absorption at 660 nm, assignable to ${}^{2}T_{2g} \rightarrow {}^{2}E_{g}$ transition. The magnetic moment of this complex is 1.72 BM at room temperature.

The spectrum of the Ni(II) complex is consistent with octahedral geometry showing two d-d transition bands at 660 and 542 nm assignable to transitions ${}^{3}A_{2g} \rightarrow {}^{3}T_{1g}(F)$ and ${}^{3}A_{2g} \rightarrow {}^{3}T_{2g}(F)$ respectively which suggests octahedral geometry. The magnetic moment values for the Ni(II) complex of the Schiff base ligand at room temperature were found to be in the normal range (2.8–3.8 B.M.).^[14] [Ni(LH₂)₂Cl₂]·2H₂O showed magnetic moment value of 5.96 BM. The high moments for the Ni(II) complex have been ascribed to quite strong ferromagnetic exchange.

The magnetic moment value for the $[Co(L)_2(AcO)_2]$ complex of the ligand is near the spin only value for high spin octahedral complexes. The electronic spectrum shows two bands at 763 and 594 nm attributed to ${}^4T_{1g} \rightarrow {}^4A_{2g}$ (v₂) and ${}^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$ (v₃) transitions respectively, in an octahedral geometry around the Co(II) ion.^[14]

The electronic spectra of the Zn(II) complex, which is diamagnetic, has bands in the 395–322 nm range and these bands may be due to the $n \rightarrow \pi^*$ and $\pi \rightarrow \pi^*$ transitions of the benzene, pyrimidine rings and azomethine group.

¹H NMR Spectra

Deuterated chloroform and deuterated DMSO were used to measure the ¹H NMR spectra of the ligand^[8] and its Zn(II) complex, respectively, except those of Cu(II), Co(II) and Ni(II) because of their paramagnetic behavior. The singlets at δ 9.0 ppm and 8.9 ppm are due to the azomethine proton of pyrimidine ring (C–H) proton in the spectrum of the ligand, respectively. In the spectra of the Schiff base the phenyl multiple was observed between δ 7.3–8.0 ppm.^[8] The ¹H NMR spectrum of the Zn(II) complex shows approximately the same peaks identical to those of the free ligand.

Conductivity

All of the complexes are non-electrolytes as shown by their molar conductivity (Λ_M) in DMF, and which are in the range^[9-12] 2.1–12.3 Ω^{-1} cm² mol⁻¹.



Thermal Studies

The thermal stability of the complexes was investigated using TGA. The TGA curves were obtained at a heating rate of 10° C/min. in N₂ atmosphere over the temperature range of $25-850.0^{\circ}$ C.

The Cu(II) complex was stable up to 30.0° C. Its decomposition started at this temperature and was completed at 692.0° C. The Cu(II) complex decomposed and produced CuO as residue [found (calculated) %: 8.30 (8.04)] in four steps in the temperature ranges 30.0-115.0, 115.0-199.0, 199.0-205.0 and $205.0-692.0^{\circ}$ C. In the decomposition process of the Cu(II) complex, the mass losses corresponded to H₂O, CH₃COO, Ph–CH– and the other organic moieties leaving in the first, second, third and fourth stages of the decomposition, respectively. The four stages of the decomposition of the Cu(II) complex were irreversible.

The Co(II) complex was stable up to 119.0°C. Its decomposition started at this temperature and was completed at 626.0°C. The Co(II) complex decomposed and produced CoO as residue [found (calculated) %: 5.81 (7.75)] in three steps in the temperature ranges 119.0–199.8, 199.8–292.0 and 292.0–626.0°C. In the decomposition process of the Co(II) complex, the mass losses corresponded to CH₃COO, Ph–CH– and the other organic moieties leaving in the first, second and third stages of the decomposition, respectively. The three stages of the decomposition of the Co(II) complex were irreversible.

The Ni(II) complex was stable up to 112.0° C. Its decomposition started at this temperature and was completed at 631.6° C. The Ni(II) complex decomposed and produced NiO as residue [found (calculated) %: 7.50 (8.12)] in two steps in the temperature ranges 112.0-210.0 and $210.0-631.6^{\circ}$ C. In the decomposition process of the Ni(II) complex, the mass losses corresponded to Ph-CH- and Cl and the other organic moieties leaving in the first and second stages of the decomposition, respectively. The two stages of decomposition of the Ni(II) complex were irreversible.

The Zn(II) complex was stable up to 172.0° C. Its decomposition started at this temperature and was completed at 629.0° C. The Zn(II) complex decomposed and produced ZnO as residue [found (calculated) %: 8.72 (8.36] in two steps in the temperature ranges 172.0-215.0 and $215.0-629.0^{\circ}$ C. In the decomposition process of the Zn(II) complex, the mass losses corresponded to Ph–CH– and the other organic moieties leaving in the first and second stages of the decomposition, respectively. The two stages of decomposition of the Zn(II) complex were irreversible.

The Cu(II) complex is thermally stable up to 30.0° C, whereas the Ni(II), Co(II), and Zn(II) complexes are thermally stable up to 112.0° C, 119.0° C, and 172.0° C, respectively. In the TGA curve of the Cu(II)

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Compounds	First step, °C	Second step,°C	Third step, °C	Fourth step, °C	(calcd.) %
$[Cu(L)_2(AcO)_2] \cdot H_2O$	30.0 - 115.0	115.0 - 199.0	199.0 - 205.0	205.0 - 692.0	CuO 8.30 (8.04)
$[Co(L)_2(AcO)_2]$	119.0 - 199.8	199.8 - 292.0	292.0 - 626.0	I	CoO 5.81 (7.75)
$[Ni(L)_2 Cl_2]$	112.0 - 210.0	210.0 - 631.6	I	I	NiO 7.50 (8.12)
$[\operatorname{Zn}(L)_2(\operatorname{AcO})_2]$	172.0 - 215.0	215.0 - 629.0	I	I	ZnO 8.72 (8.36)

91.70 (91.96) 94.19 (92.25) 91.88 (91.88) 91.28 (91.64) Total 59.65 (60.04) Fourth step Table 4. TGA data of the complexes [weight loss %, found (calculated)]. I 1 1 17.94 (18.19) 64.60 (63.08) Third step I. 12.28 (11.92) 19.09 (18.62) 72.28 (72.31) 73.08 (73.15) Second step 1.83 (1.81) 10.50 (12.20) 19.60 (19.57) 18.20 (18.49) First step $[Cu(L)_2(AcO)_2] \cdot H_2O$ [Ni(L)₂Cl₂] [Zn(L)₂(AcO)₂] $[Co(L)_2(AcO)_2]$ Compounds

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Figure 2. Proposed structure for all the complexes.

complex, 1.83% weight loss was observed at 115.0° C. This shows that the complex contains 1 mole of water of crystallization per complex molecule. The IR spectra of the complexes show a broad band in the region $3230-3650 \text{ cm}^{-1}$ due to the v(O–H) frequency of water of crystallization.^[6,13] This water content was also identified by elemental analyses. The decomposition process of the Cu(II), Ni(II), Co(II) and Zn(II) complexes was also confirmed by TGA data. As can be seen in Table 3, the most stable complex is the Zn(II) complex.

The weight losses for the Cu(II), Ni(II), Co(II) and Zn(II) complexes were found to be approximately the same, when expressed as the percentages calculated stoichiometrically from their chemical formulas given in Tables 1, 3 and 4. All of these complexes undergo complete decomposition to the corresponding metal oxides, CuO, NiO, CoO or $ZnO^{[6,15-18]}$ (Table 3).

Single crystals of the complexes could not be isolated from any solutions, thus no definitive structure could be described. However, the analytical, spectroscopic and magnetic data enable us to propose the possible structures as shown in Figure 2.

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