ORIGINAL ARTICLE

Synthesis, Spectroscopic Study and Biological Activity of new Bidentate Schiff Base (4-(Dimethylamino) Benzylidene)-5-(3,4,5-Trimethoxybenzyl)-5 Pyrimidine-2,4-Diamine) and Transition Metal Mn(II), Ni(II) and Cu(II) Complexes

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ABSTRACT

The Schiff bases ligand (L₁) was synthesized by condensing (2,4-diamine (Trimethoxybenzyl 3,4,5)-5 pyrimidine) and (4dimethylamino benzaldehyde). The transition metal complexes of $[Mn(L_1)_2Cl_2]$, $[Ni(L_1)_2Cl_2]$, and $[Cu (L_1)_2Cl_2]$ were then produced. The ligand and its metal complexes Mn(II), Ni(II), and Cu(II) were examined by analyzing the elemental composition, the metal content, the chloride content, and the molar conductance, FT-IR, 1H-NMR, UV-Vis spectra, magnetic susceptibility, mass spectra, and thermal analysis (TGA). The results indicated that the ligand behaves as a bidentate ligand in all of the complexes produced. The [Cu (L₁)₂Cl₂] complex is more poisonous to microorganisms. **Keywords:** Ligand field, Schiff bases, Mass spectra, Charge transfer (C.T).

INTRODUCTION

Trimethoprim, chemical formula (C14H19N4O3), systematically named as (2,4-diamine (Trimethoxybenzyl 3, 4,5)-5 pyrimidine), its white to yellowish compound with bitter taste, it contains two 3,4,5-trimethoxytoluene components: and 2,4-diamino-5methylpyrimidine. It is a dihydrofolate reductase inhibitor, which is a class of chemotherapeutic medicines. It is useful in the treatment of prevention and urinary tract infections [1]. Aroylhydrazones can operate as N4 chelators, forming redox-efficient complexes that cause reactive oxygen species generation, inhibit metalloproteins, interact with DNA, and alter intracellular homeostasis [2]. Trimethoprim and its derivatives are antimicrobials with antiparasitic action that were discovered by Roth and coworkers [3]. Due to their anticancer, antibacterial, antiviral, and antifungal activities, due to the presence of the -C=N- group, Schiff bases are often referred to as azomethine. They serve a key function in biological systems. They have also been extensively used as ligands in coordination chemistry due to their superior donor properties as chelating agents [3-7]. Additionally, Schiff bases are widely used as catalysts and photochromic sensors in a variety of fields of life sciences [8]. Due to their stability, ease of modification, and huge biological characteristics, Schiff base-transition metal complexes have garnered considerable attention in recent years [9, 10]. In this respect, The Schiff base ligand was produced and characterized by a series of reactions (2,4-diamine (Trimethoxybenzyl 3,4,5)-5 pyrimidine) with (4-dimethylamino benzaldehyde) and their metal complexes. Experimental

Materials and measurements: All chemicals and reagents were obtained commercially (Sigma-Aldrich, Merck, etc.) and used unpurified. The 1H-NMR spectra were acquired using the following techniques: Bruker On Eurovector EA 3000A, Al al-Bayt University, Ultra Sheild 300 MHz NMR and elemental microanalysis (C.H.N) were done (Jordan). The metal ion content was determined gravimetrically as metal oxides. Conductometer WTW was used to measure the molar conductance of metal complexes of the ligand at a concentration of 1x10-3M. Magnetic measurements were made at 25 °C, England, by the Johnson mattey catalytic system division of balance. UV-Vis spectra were examined using a Shimadzu UV-1800 spectrophotometer. using a quartz cell 1.0 cm from the wavelength range (200-1100 nm). SHIMADZU FT-IR 8400S was used to acquire Fourier Transform Infrared (FT-IR) spectra. Fourier transformations in the wavenumber range 4000 to 200 cm-1 using KBr and CsI discs, Department of Chemistry, College of Science, University of Baghdad. The mass spectra were acquired on the GC MS -QP 2010 VLTRA instrument at the Department of Chemistry, College of Science, AL- Mustansiriyah University. Thermo gravimetric analyses were carried out experimentally using Perkin-Elmer Pyris Diamond TG. All experiments were conducted in a single crucible with a flow rate of 10oC/min and a temperature range of 25-700oC. Biological activities of selected samples were determined using the agar diffusion method developed by the Ministry of Science and Technology.

Synthesis of ligand (L1): 4-(dimethylamino)benzylidene)-5-(3,4,5-trimethoxybenzyl)-5 pyrimidine-2,4-diamine: This ligand was synthesized by reacting (3.5 mmol, 1 g) (2,4-diamine (Trimethoxybenzyl 3,4,5)-5 pyrimidine) and (3.5 mmol, 0.5 g) (4dimethylamino benzaldehyde) in a 30 ml pure alcoholic medium with the addition of 5 drops of acetic acid glacial. The mixture was then refluxed for 8 hours to precipitate the product The product was ethanol crystallized -ether (1:1), collected, and dried in vacuum over anhydrous CaCl2. The melting point of the ligand is determined to be (228-230 oC), the molecular weight is (421.49 g mol-1), and the yield is 76 percent. (Scheme-1.a) The ligand's synthesis pathway. The ¹HNMR spectrum of ligand L₁ in DMSO-d6 is shown in (Figure -1). The spectrum displayed a peaks observed at δ (2.328-2.882) ppm was assigned to solvent DMSO, peaks observed at δ (3.235) (2H) s , (CH₂), δ (3.535-4.216) (9H) m, (OCH₃), δ (4.330-4.599) (6H) t , (N-CH₃), δ (6.646-7.412) (7H) m arom , δ(8.292) (2H) s, (NH₂), 9.195 (1H) s azomethine group (N=C-H) [3,4,5]

Procedures for the production of metal complexes in general: It was prepared by dissolving Schiff base ligand L_1 an ethanolic solution (2mol) (30 ml) in around beaker, then, added (NaOH + water) with stirring after that added metal salt (1mol) that dissolve in ethanol– water (1:1) (20 ml). The combination was heated for 4 hours while stirring, the precipitate and solution were filtered, the product was ethanol was used to clean the crystals, and they were recrystallized in ethanol and diethyl ether. The product was collected and dried in vacuum over anhydrous CaCl2. (Scheme-1.b) Metal complexes with proposed structures.



Scheme-1A: L1 ligand synthesis pathway



Scheme -1B: Structures of metal complexes that have been proposed



Figure 1: ¹H-NMR spectrum of ligand L₁

RESULTS AND DISCUSSIONS

The bi-identical complexes were synthesized by reacting metal ions in a 1:2 molar ratio with a Schiff base (metal:ligand). The stiff composite base Schiff ligands and complexes are extremely stable at room temperature. Additionally, there are physicochemical data given in (Table 1).

Table 1: The elements' results of rigorous research and certain physical features of the L1 bond and their metal complexes

Compound	Formula	M.wt	Elemental Anal	Elemental Analysis Cal (Found)%					
			С	Н	N	М	CI		
L ₁	C ₂₃ H ₂₇ N ₅ O ₃	421.49	(65.12)	(6.33)	(17.30)	-	-		
			65.54	6.46	16.62				
[Mn (L1)2Cl2]	C46H54N10O6MnCl2	967.13	(56.48)	(5.10)	(13.94)	(5.60)	(7.11)		
			57.07	5.58	14.47	5.67	7.32		
[Ni (L1)2Cl2]	C46H54N10O6NiCl2	970.89	(57.44)	(5.28)	(14.10)	(5.83)	(7.20)		
			56.85	5.56	14.41	6.04	7.29		
[Cu (L ₁) ₂ Cl ₂]	C ₄₆ H ₅₄ N ₁₀ O ₆ CuCl ₂	975.74	(54.93)	(5.21)	(14.45)	(6.33)	(7.03)		
			56.57	5.53	14.34	6.51	7.25		

Mass spectrum: Mass spectra of the new ligand were obtained using electron impact of fragmentation [11, 12].

In Figure 1, The effect of the electron effect on the mass spectrometry of the L₁ ligand is depicted (Figure -2). This ligand has an estimated molecular weight of (421.49) g/mol. The spectrum revealed a peak at (421) m/z corresponding to the Schiff base moiety [$C_{23}H_{27}N_5O_3$], which was attributed to [M]+. Other distinctive peaks at (254, 167, 134, and 120) m/z may correspond to other pieces. Their intensity conveys information about the shards' stability. The proposed paths for fragmentation and the structural assignments of fragments are outlined in (Scheme -2).



Figure 2: The mass spectrum of the L1 ligand as determined by LC.



Scheme 2: L_1 ligand fragmentation pattern

Infrared analysis: The FT-IR spectra of the ligand L_1 and the complex Ni(II) L_1 is shown in (Figures -3,4). The linker (L_1) and its mineral complexes absorbance bands are summarized in (Table-2). All compounds' infrared spectra revealed associative bands with proper transitions according to their complex architecture. These findings suggest that ligand L_1 couples with the metal ion via the azomethine nitrogen groups and the nitrogen pyrimidine ring, which functions as a double bond [11, 13-18].

Sym.	V(NH ₂)	v(C-H) Arom.	v(C-H) aliph.	V(C=N)	v(C=C) Arom.	V(C=N)	∨ (M-N)	V (M-CI)
				azomethine		Pyrimidine		
L ₁	3394.72	3257.17	2932.09	1634.35	1446.61	1527.62	-	-
Mn L ₁	3452.17	3045.22	2954.56	1649.33	1459.81	1577.32	546.12	478.64
Ni L ₁	3421.01	3178.68	2991.59	1651.07	1498.68	1595.13	534.28	495.71
Cu L ₁	3413.47	3073.43	2973.46	1653.51	1477.65	1568.41	525.01	453.63

Table 2: The ligand (L₁) and its metal complexes infrared spectral data





Figure 4: FT.IR Spectrum of [Ni(L1)2Cl2]complex

Electronic spectra and magnetic moments studies: The UV-V spectra of ligand L₁ and its compounds provide insight into the (Table-3) Demonstrate a tape with a high density for optimal absorption at λ 270 nm (v_{max} 37037.0 cm⁻¹, ϵ_{max} 2355 L.mol⁻¹.cm⁻¹) attributed to the $\pi \rightarrow \pi^{\star}$ transition, and peak at λ 355 nm (v_{max} 28169.0 cm⁻¹, ϵ_{max} 1735 L.mol⁻¹.cm⁻¹) attributed to the $n \rightarrow \pi^{\star}$ transition, in this case, the azomethine group may be assigned to the $n \rightarrow \pi^{\star}$ transition. The shift of this band in the complexes' spectra revealed that the nitrogen was coordinated to the metal atom.

Manganese(II) complex: At ambient temperature, the Mn(II) complex has a magnetic moment of 5.02 BM, which corresponds to the five unpaired electrons. Four absorption peaks were seen in the electronic spectra of the Mn L_1 complex. The two peaks at (282, 345) and (C. T), respectively, are attributed to the ligand field and (C. T), respectively. The final two peaks at (520, 710) nm are attributed to (d-d)electronic transitions, which provide strong support for Octahedral geometry.

Nickel (II) complex: Five absorption peaks are visible in the absorption spectrum of Ni(II) complexes, The two peaks at (275, 332) nm attributed to the ligand field and (C. T), the three d–d transition bands at (36383.6, 30120.4, 21276.5, 16949.1 and 15037.5 cm⁻¹ which correspond to ${}^{3}A_{2}g(F) \rightarrow {}^{3}T_{1}g(P)$, ${}^{3}A_{2}g(F) \rightarrow {}^{3}T_{1}g(F)$ and ${}^{3}A_{2}g(F) \rightarrow {}^{3}T_{2}g(F)$. Magnetic moments of Ni (II) complexes were determined to be 2.85 BM, consistent with a deformed octahedral structure with d8 high spin.

Copper (II) complex: The absorption spectra of Cu(II) complex display three absorption peaks, The two peaks at (285, 340) nm attibuted to the ligand field and (C. T), the last peak attributed to d– d transition ${}^{2}\text{Eg} \rightarrow {}^{2}\text{T}_{2}\text{g}$. Cu(II) complexes were discovered to have magnetic moments of 1.82 BM, indicating an octahedral shape. While the low conductivity values indicated that the complexes were not electrolytes, they also indicated that the chloride ion remained coordinated to the metal ion [19-24].

Table 3: Electronic S	pectral Data on Metal Co	nplexes including) (L1)) Ligand,	Molar Conductivit	y in	(DMSO 10-3M), and Mag	gnetic Moments
				, ,				,,	

Complex	λ _{max}	v cm ⁻¹	ABS	ε _{max} L	Assignment	Δm cm ²	μ _{eff}
Geometry	(nm)			mol ⁻¹ cm ⁻¹	-	Ω ⁻¹ mol ⁻¹	B.M
L ₁	270	37037.0	2.355	2355	$\pi \rightarrow \pi^*$	-	-
	355	28169.0	1.735	1735	n→π*		
[Mn(L ₁) ₂ Cl ₂]	282	35460.9	1.853	1853	L.F		
Oh	345	28985.5	2.435	2435	C.T	12.08	5.02
	520	19230.7	0.875	875	${}^{6}A_{1g} \rightarrow {}^{4}T_{2g} (G)$		
	710	14084.5	0.533	533	$^6A_{1g} \rightarrow {}^4T_{2g(F)}$		
[Ni (L ₁) ₂ Cl ₂]	275	36383.6	1.805	1805	L.F		
Oh	332	30120.4	2.107	2107	C.T	10.50	2.85
	470	21276.5	0.932	932	${}^{3}A_{2g(F)} \rightarrow {}^{3}T_{1g(P)}$		
	590	16949.1	0.455	455	${}^{3}A_{2g(F)} \rightarrow {}^{3}T_{1g(F)}$		
	665	15037.5	0.123	123	$^{3}A_{2g(F)} \rightarrow ^{3}T_{2g(F)}$		
[Cu(L ₁) ₂ Cl ₂]	285	35087.7	1.745	1745	L.F	19.10	1.82
Oh	340	29411.7	2.082	2082	C.T		
	495	20232.2	0.627	627	² Eg→ ² T ₂ g		

Oh= Octahedral

Table 4: Thermal decomposition of ligand L_1 and their $[Cu(L_1)_2Cl_2]$ complex

Comp.	Thermogra-vimetric range (°C)	DTG max(°C)	% Estimated (calculated)		Assignment
			Mass loss	Total Mass loss	
ligand L1	40-110 110-430 430-600	100.265 392.662 594.884	6.1081 (7.3548) 31.7361 (32.2664) 45.8984 (46.0271) 16.2580 (15.2352)	83.7426 (85.6483)	OCH3 2OCH3+ C6H2 C9H16N5 Residue C5
[Cu(L ₁) ₂ Cl ₂]	75-230 230-600	215.666 594.549	15.541 (15.8853) 73.8985 (73.4826) 10.5605 (10.6073)	89.4395 (89.3679)	$\begin{array}{c} \text{5OCH}_3\\ \text{C}_{39}\text{H}_{38}\text{N}_{10}\text{CI}_2\\ \text{Residue}\\ \text{CuO} + \text{C}_2 \end{array}$

Thermal Analysis Data: The thermal decomposition of ligand L_1 and its complex [Cu(L₁) $_2$ Cl₂] using TG and DTG is discussed in

depth in (Table-4) (Figs-5-6). To gain a better understanding of the thermal decomposition process, the Schiff ligand L_1 base and its

mineral complex were investigated using thermogravimetric analysis at temperatures ranging from 35 to 700 degrees Celsius at a heating rate of 10 degrees Celsius per minute in a nitrogen atmosphere. The thermograms were used to compute the complexes' breakdown phases, they demonstrated agreement in weight loss between the amounts received via heat. [25,26].



Figure 5: TG ligand thermogravimetric analysis (L₁)



Figure 6: TG thermo gravimetric of [Cu(L1)2Cl2]

Antibacterial Activates: The nutrient agar plates were prepared and incubated overnight at 37 °C to check for bacterial contamination. The bacterial suspension was grown in nutrient agar at 37°C. Then each compound spotted on the nutrient media followed by incubation for 18h at 37°C. Then inhibition zone diameters (mm) were measured for each product relate to the blank solvent DDW. The results indicate that the complexes are more hazardous to bacteria than the free ligand. This is consistent with the tweeds chelation idea. Compounds' mode of action may entail the interaction of their (C=N), (CH3), (OCH₃), and (NH₂) groups with the active centers of cell constituents, thereby interfering with normal cell processes [27-30]. Antibacterial activities of the ligand (L₁) and their metal complexes are explained in the (Table -5).

Table 5: The ligand (L1) and its mineral complexes has antibacterial properties.

No.	Comp.	Inhibition							
		Diameter (mm)							
		Escherichia coli	Streptococcus pneumonia	Pseudomonas aeruginosa	Staphylococcus aureus				
1.	TRM	20 mm	25 mm	10 mm	18mm				
2.	L ₁	32mm	30mm	8mm	37mm				
3.	MnL ₁	35mm	30mm	9mm	35mm				
4.	NiL ₁	32mm	28mm	8mm	33mm				
5.	CuL ₁	36mm	40mm	14mm	38mm				

CONCLUSION

This paper, new Mn(II), Ni(II) and Schiff base ligand (L₁) Cu(II) complexes (4-(dimethylamino)benzylidene)-5-(3,4,5-trimethoxybenzyl)-5 pyrimidine-2,4-diamine). Physicochemical and spectroscopic approaches were used to determine the bonding mode and The complexes' general structure. The spectroscopic investigation of In all complexes, the ligand L₁ is bidentate ligand and The azomethine nitrogen groups and the nitrogen pyrimidine ring coordinate metal ions. Based on UV-vis and FT-IR spectra, it was proposed that the geometry of all metal complexes is octahedral. The CuL₁complex have more toxicity against the bacterial species than the free ligand. (Figure -7) The 3D chemical structures of the ligand L₁.



Figure 7: The 3D chemical structures of the ligand L1

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