

SYNTHESIS, SPECTROSCOPY, AND ANTIBACTERIAL ACTIVITY OF SOME TRANSITION METAL COMPLEXES WITH TRIDENTATE (ONS) AND BIDENTATE (NN) DONOR SCHIFF BASES

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The complexes of a series of transition metal ions including Mn(II), Co(II), Ni(II), Cu(II), Zn(II), and Cd(II) with salicylidene-*o*-aminothiophenol and bis(benzylidene)ethylenediamine or bis(acetophenone)ethylenediamine have been synthesized and characterized by elemental analyses, infrared spectra, reflectance spectra, magnetic measurements, and thermogravimetric analyses. All complexes have been screened for their antibacterial activity against bacterial strains using the disc diffusion technique. It is established that the complexes possess moderate antibacterial activity against *Salmonella typhimurium*, *Escherichia coli* and *Serratia marcescens*, the effect being higher as compared to the action of individual ligands, metal chlorides, and control (DMSO), but lower than the action of the reference drug tetracycline.

A large number of Schiff bases and their complexes were studied for their interesting and important properties, such as the ability to reversibly bind oxygen [1], catalytic activity in the hydrogenation of olefins [2], transfer of amino group [3], photochromic properties [4], and complex-forming ability with respect to some toxic metals. Metal complexes of Schiff bases derived from substituted salicylaldehyde and various amines have been extensively investigated [5]. However, little attention has been given to Schiff bases containing ONS and NN donor systems. In continuation of our earlier work [6], we report here the method of synthesis and the results of characterization and antibacterial activity evaluation for the complexes of some transition metal ions with salicylidene-*o*-aminothiophenol and bis(benzylidene)ethylenediamine or bis(acetophenone)ethylenediamine.

The elemental compositions, empirical formulas, molecular weights, colors, percentage yields, melting points and magnetic moments are presented in Table 1. The data of elemental analyses for Schiff bases H₂L, SB¹, SB² and their complexes are in good agreement with the results of calculations performed using the proposed formula.

The IR spectra of the synthesized complexes were compared to those of the initial Schiff bases H₂L, SB¹, and SB² in order to determine the coordination sites involved in chelation. The OH stretching frequency $\nu(\text{OH})$ [7] is present as a medium intensity band at 3200–3300 cm⁻¹ in the spectra of Schiff bases, but it disappears in the spectra of complexes thus indicating the participation of the OH group in chelate formation via proton displacement. On the other hand, the band at ~3400 cm⁻¹ in the spectra of complexes is indicative of the presence of coordination water [8]. Thus, it is difficult to draw conclusions based on the $\nu(\text{OH})$ frequency of Schiff bases. However, this OH vibration disappeared on drying the complexes at 200°C, which is evidence for the participation of these hydroxy groups in chelation. The bands at ~850 cm⁻¹ and ~715 cm⁻¹ are assigned to the rocking and wagging vibrations of OH groups. Sharp bands in the regions of 750–780 cm⁻¹ and 1525–1535 cm⁻¹ are due to aromatic $\nu(\text{C-H})$ and $\nu(\text{C=C})$ vibrations, respectively. The $\nu(\text{SH})$ stretching vibration is not helpful since it gives only very weak bands in the spectra of both free ligands and their complexes. However, the participation of SH groups in chelation is confirmed by a shift of the $\nu(\text{C-S})$ band to lower frequen-

cies [9], from 765 cm⁻¹ (in the free ligand) to ~750 cm⁻¹ (in the complex). The $\nu(\text{C=N})$ stretching vibration, which is found for the free ligand at 1625 cm⁻¹ and shifts to a lower frequency (about ~1610 cm⁻¹) in the complexes, points to the participation of azomethine nitrogen [10] in coordination. The IR bands of medium intensity at ~440 and ~520 cm⁻¹ are attributed to $\nu(\text{M-O})$ [11] and $\nu(\text{M-N})$ [12], respectively. The complexes also show a new band in the region of 410–425 cm⁻¹, which can be assigned to $\nu(\text{M-S})$ [13]. The bands in the regions of 1470–1480 and 1370–1380 cm⁻¹ are due to $\nu(\text{C-H})$ of alkane and $\nu(\text{C-CH}_3)$, respectively. The absorption in the range 1160–1170 cm⁻¹ is attributed to $\nu(\text{C-N})$.

The UV-VIS spectra provide the most detailed information about the electronic structure. The reflectance spectra of complexes were studied in solid state samples. The spectra of Ni(II) complexes show three bands (at ~10,500, ~18,000, and ~24,000 cm⁻¹), which can be due to the ³A_{2g}(F) → ³T_{2g}(F) (ν_1), ³A_{2g}(F) → ³T_{1g}(F) (ν_2) and ³A_{2g}(F) → ³T_{1g}(P) (ν_3) transitions, respectively, in an octahedral field of Ni(II) ions [14]. The magnetic moments of Ni(II) complexes are equal to 2.78 and 2.80 B. M., which are typical of similar hexacoordinated Ni(II) ions.

The Co(II) complexes show three bands at ~9,200 (ν_1), ~17,900 (ν_2) and ~19,000 (ν_3) cm⁻¹, which may be assigned to the ⁴T_{1g}(F) → ⁴T_{2g}(F), ⁴T_{1g}(F) → ⁴A_{2g}(F) and ⁴T_{1g}(F) → ⁴T_{1g}(P), transitions expected for an octahedral structure [15]. The magnetic moment of Co(II) complexes amount to 4.01 and 4.00 B. M., which is somewhat higher than the expected value (3.87 B. M.). This increase can be due to incomplete quenching of the orbital magnetic moment by the surrounding ligands, suggesting an octahedral geometry around the Co(II) ion.

The electronic spectra of Cu(II) complexes exhibit a single band at ~15,500 cm⁻¹, which can be assigned to ²E_g → ²T_{2g} transition in an octahedral geometry [16]. The magnetic moment of Cu(II) complexes were observed in the range of 1.78–1.81 B. M., which corresponds to a single unpaired electron with a very slight orbital contribution and suggests an octahedral geometry around the Cu(II) ion.

The electronic spectra of Mn(II) complexes exhibit three weak absorption bands at ~15,000, ~19,500 and ~24,500 cm⁻¹, which may be assigned to transitions ⁶A_{1g} → ⁴T_{1g}(⁴G)

(v_1), ${}^6A_{1g} \rightarrow {}^4T_{2g}({}^4G)$ (v_2), and ${}^6A_{1g} \rightarrow {}^4E_g, {}^4A_{1g}({}^4G)$ (v_3), respectively, in an octahedral field of Mn(II) ions [17]. The magnetic moments of Mn(II) complexes are 6.00 and 6.02 B. M., which fall within the interval of spin-free values for five unpaired electrons, thus indicating that the complexes are high-spin d^5 systems with an octahedral structure.

The Zn(II) and Cd(II) complexes are diamagnetic, as expected for d^{10} systems.

Thermogravimetric analyses of the complexes were carried out in the temperature range of 50 – 800°C. It was found that all complexes show a weight loss corresponding to one water molecule in the range ~150 – 180°C, indicating that this water molecule are coordinated to the metal ion [18]. In the temperature range 180 – 800°C, the ligand molecules are lost. In all cases, the final products are metal oxides. These results are in good accordance with the proposed compositions of complexes.

Experimental chemical part

The magnetic moments were measured using a Model 7304 vibrating sample magnetometer (Lake Shore, USA). Infrared spectra were recorded on a Nicolet 400D FTIR spectrophotometer. Carbon, hydrogen, nitrogen and sulfur were analyzed with a Perkin-Elmer Model 240 elemental analyzer.

The metal contents of the complexes were analyzed using the EDTA titration technique [19] after decomposition of the organic matter with a mixture of perchloric, sulfuric, and nitric acid (1 : 1.5 : 2.5). Thermogravimetric analyses were performed on a Model 5000/2960 analyzer (T. A. Instruments, USA). The reflectance spectra of complexes were recorded in the range from 1700 to 350 nm (as MgO discs) on a Beckman DK-2A spectrophotometer.

Salicylidene-*o*-aminothiophenol (H₂L) was prepared by a condensation reaction between salicylaldehyde (10 mmol, 1.22 g) and *o*-aminothiophenol (10 mmole, 1.25 g) in ethanol (100 ml). The precipitate was filtered and recrystallized from dilute acetic acid. The structure was confirmed by elemental analyses and IR spectra. Yield, 70 %; m.p., 128°C. The structure of H₂L is shown in Fig. 1.

Bis(benzylidene)ethylenediamine (SB¹) was synthesized as described in [20].

Bis(acetophenone)ethylenediamine (SB²). Ethanol solutions of ethylenediamine (10 mmole, 0.60 g) and acetophenone (20 mmole, 2.40 g) in ratio of 1 : 2 were mixed with constant stirring and reflux for 5 h and cooled in refrigerator to 0 – 2°C overnight. The precipitated yellow crystals were collected and dried in air. Yield, 76 %; m.p., 260°C. The pro-

Table 1

Yields and Physicochemical Characteristics of Complexes and Their Components

Compounds empirical Formula	Mol. Wt.	Colour	%Found (Cal.)					M.p., (°C)	μ_{eff} (B.M.)	% Yield
			C	H	N	S	Metal			
H ₂ L	229.21	Yellow	68.06	4.80	6.07	13.92	–	128	–	70
C ₁₃ H ₁₁ NOS			(68.11)	(4.79)	(6.10)	(13.99)				
SB ¹	236.16	Yellow	81.32	6.78	11.80	–	–	235	–	72
C ₁₆ H ₁₆ N ₂			(81.36)	(6.77)	(11.85)					
SB ²	264.18	Yellow	81.80	7.55	10.64	–	–	260	–	76
C ₁₈ H ₂₀ N ₂			(81.83)	(7.57)	(10.60)					
[Mn(L)(SB ¹)(H ₂ O)]	536.28	Brown	64.90	5.00	7.80	5.94	10.20	>360	6.00	40
C ₂₉ H ₂₇ MnN ₃ O ₂ S			(64.94)	(5.03)	(7.83)	(5.97)	(10.24)		(5.92)	
[Co(L)(SB ¹)(H ₂ O)]	540.28	Green	64.45	4.95	7.81	5.94	10.96	>360	4.01	35
C ₂₉ H ₂₇ CoN ₃ O ₂ S			(64.46)	(4.99)	(7.77)	(5.93)	(10.90)		(3.87)	
[Ni(L)(SB ¹)(H ₂ O)]	540.04	Light green	64.52	5.03	7.75	5.90	10.90	155	2.78	40
C ₂₉ H ₂₇ NiN ₃ O ₂ S			(64.49)	(4.99)	(7.77)	(5.93)	(10.86)		(2.83)	
[Cu(L)(SB ¹)(H ₂ O)]	544.89	Green	63.88	5.00	7.67	5.92	11.60	260	1.78	42
C ₂₉ H ₂₇ CuN ₃ O ₂ S			(63.91)	(4.95)	(7.70)	(5.88)	(11.64)		(1.73)	
[Zn(L)(SB ¹)(H ₂ O)]	546.74	Yellow	63.72	4.97	7.65	5.85	11.93	>360	–	55
C ₂₉ H ₂₇ ZnN ₃ O ₂ S			(63.70)	(4.93)	(7.68)	(5.86)	(11.96)			
[Cd(L)(SB ¹)(H ₂ O)]	593.76	Yellow	58.69	4.50	7.11	5.42	19.00	>360	–	42
C ₂₉ H ₂₇ CdN ₃ O ₂ S			(58.65)	(4.54)	(7.07)	(5.39)	(18.93)			
[Mn(L)(SB ²)(H ₂ O)]	564.24	Yellow	66.01	5.20	7.03	5.40	9.70	>360	6.02	36
C ₃₁ H ₃₁ MnN ₃ O ₂ S			(65.98)	(5.22)	(7.07)	(5.39)	(9.73)		(5.92)	
[Co(L)(SB ²)(H ₂ O)]	568.24	Brown	65.53	5.41	7.42	5.61	10.42	>360	4.00	35
C ₃₁ H ₃₁ CoN ₃ O ₂ S			(65.51)	(5.45)	(7.39)	(5.64)	(10.37)		(3.87)	
[Ni(L)(SB ²)(H ₂ O)]	568.00	Reddish	65.59	5.48	7.35	5.69	10.30	150	2.80	41
C ₃₁ H ₃₁ NiN ₃ O ₂ S		Yellow	(65.54)	(5.45)	(7.39)	(5.64)	(10.33)		(2.83)	
[Cu(L)(SB ²)(H ₂ O)]	572.65	Light green	65.03	5.40	7.34	5.57	11.08	250	1.80	45
C ₃₁ H ₃₁ CuN ₃ O ₂ S			(65.01)	(5.41)	(7.33)	(5.50)	(11.09)		(1.73)	
[Zn(L)(SB ²)(H ₂ O)]	574.70	Yellow	64.75	5.35	7.32	5.55	11.40	>360	–	57
C ₃₁ H ₃₁ ZnN ₃ O ₂ S			(64.78)	(5.39)	(7.30)	(5.60)	(11.37)			
[Cd(L)(SB ²)(H ₂ O)]	621.72	Yellow	59.85	4.90	6.73	5.20	18.10	>360	–	44
C ₃₁ H ₃₁ CdN ₃ O ₂ S			(59.88)	(4.85)	(6.75)	(5.15)	(18.08)			

H₂L = salicylidene-*o*-aminothiophenol, SB¹ = bis(benzylidene)ethylenediamine, SB² = bis(acetophenone)ethylenediamine

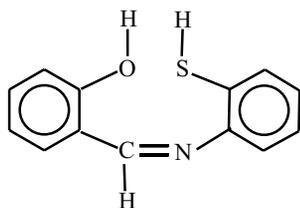


Fig. 1. The structure of salicylidene-*o*-aminothiophenol (H_2L).

posed structures of bis(benzylidene) ethylenediamine and bis(acetophenone)ethylenediamine are shown in Fig. 2.

Synthesis of complexes. A mixture of 100 ml of an aqueous solutions of metal chloride (10 mmole) and hot methanol solutions of salicylidene-*o*-aminothiophenol (H_2L) (10 mmole, 2.29 g) and bis(benzylidene)ethylenediamine (SB^1) (10 mmole, 2.36 g) or bis(acetophenone)ethylenediamine (SB^2) (10 mmole, 2.64 g) in 1 : 1 : 1 molar ratio was heated on a water bath for 2 h at 50°C and then kept overnight at room temperature. The precipitated crystals were separated by filtration, washed with water and ethanol, and dried in air.

Experimental biological part

The antibacterial activity of the control substance (dimethylsulfoxide, DMSO), the reference drug (tetracycline), individual metal chlorides and ligands, and their complexes were evaluated using the disc diffusion method [21]. A stock solution of 250 $\mu\text{g/ml}$ was prepared by dissolving 25 mg of each compound in DMSO (100 ml). Filter paper (Whatmann No. 4) 6-mm-diameter discs were impregnated with solutions

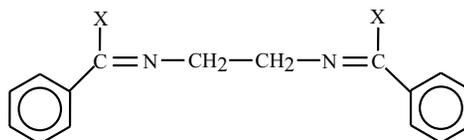


Fig. 2. The structures of bis(benzylidene)ethylenediamine (SB^1 , $X = H$) and bis(acetophenone)ethylenediamine (SB^2 , $X = CH_3$).

(20 μl) of test compounds, dried, and placed on a nutrient agar plate containing lawn cultures of various bacteria. The plates were incubated at an optimum growth temperature (37°C) for 24 h and then the zone of microbial growth inhibition around the discs was measured (in mm). The test bacteria included (a) *Salmonella typhimurium*, (b) *Escherichia coli*, and (c) *Serratia marcescens*. The control experiments were performed where only equivalent volume of solvents without added test compounds were applied onto the paper discs. All experiments were performed in triplicate and tetracycline was used as a reference drug. Since the complexes were dissolved in DMSO, the microbial growth was compared to that in DMSO (control), and the size of the inhibition zone was evaluated as percentage versus control.

Results and discussion

Previously, antitumor [22], antiviral [23], and antimalarial properties [24] were studied for various coordination compounds obtained using the ability of metal ions to form stable complexes [25]. The results elucidated the coordination sphere, the electronic properties of metal ions, and some other factors such as chelate formation, ring size, number of aro-

Table 2

Results of Tests for Antibacterial Activity

Compounds	Zone of inhibition (in mm) (% inhibition)		
	<i>S. typhi</i>	<i>E. coli</i>	<i>S. marcescens</i>
DMSO (control)	9	12	15
Tetracycline	18 (100.00)	21 (75.00)	26 (73.33)
H_2L	11 (22.22)	14 (16.66)	17 (13.33)
SB^1	11 (22.22)	14 (16.66)	17 (13.33)
SB^2	11 (22.22)	13 (08.33)	16 (06.66)
$MnCl_2 \cdot 4H_2O$	10 (11.11)	13 (08.33)	16 (06.66)
$CoCl_2 \cdot 6H_2O$	10 (11.11)	13 (08.33)	17 (13.33)
$NiCl_2 \cdot 6H_2O$	11 (22.22)	14 (06.66)	16 (06.66)
$CuCl_2 \cdot 2H_2O$	11 (22.22)	13 (08.33)	17 (13.33)
$ZnCl_2$	10 (11.11)	14 (16.66)	17 (13.33)
$CdCl_2 \cdot 2H_2O$	11(22.22)	14 (16.66)	17 (13.33)
$[Mn(L)(SB^1)(H_2O)]$	14 (55.55)	15 (25.00)	19 (26.66)
$[Co(L)(SB^1)(H_2O)]$	13 (44.44)	16 (33.33)	21 (40.00)
$[Ni(L)(SB^1)(H_2O)]$	13 (44.44)	15 (25.00)	19 (26.66)
$[Cu(L)(SB^1)(H_2O)]$	12 (33.33)	14 (16.66)	20 (33.33)
$[Zn(L)(SB^1)(H_2O)]$	14 (55.55)	18 (50.00)	22 (46.66)
$[Cd(L)(SB^1)(H_2O)]$	12 (33.33)	17 (41.66)	20 (33.33)
$[Mn(L)(SB^2)(H_2O)]$	12 (33.33)	17 (41.66)	20 (33.33)
$[Co(L)(SB^2)(H_2O)]$	13 (44.44)	15 (25.00)	19 (26.66)
$[Ni(L)(SB^2)(H_2O)]$	13 (44.44)	16 (33.33)	21 (40.00)
$[Cu(L)(SB^2)(H_2O)]$	14 (55.55)	18 (50.00)	23 (53.33)
$[Zn(L)(SB^2)(H_2O)]$	13 (44.44)	17 (41.66)	22 (46.66)
$[Cd(L)(SB^2)(H_2O)]$	14 (55.55)	16 (33.33)	20 (33.33)

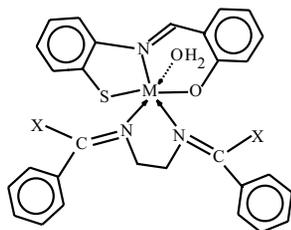


Fig. 3. Suggested structure of the mixed-ligand complexes.



matic rings, and the presence of amino groups, which modify the coordination sphere. It was suggested that heterocyclic rings containing sulfur, nitrogen, and/or oxygen are responsible for the biological activity of the Schiff bases and related metal complexes [26]. Furthermore, it has been demonstrated that chelation in these compounds, is also responsible to a large extent, for such activity [27].

Bases on the above structural considerations, the octahedral structure (Fig. 3) has been tentatively proposed for all the synthesized mixed-ligand complexes.

Data on the zone of inhibition and percentage inhibition of the test microbe growth for the synthesized complexes are presented in Table 2. The results of disc diffusion tests show that mixed-ligand complexes are more toxic than the solvent (control) and the individual ligands and metal salts against the same microorganisms under identical experimental conditions. The increase in antibacterial activity of the metal chelates may be due to the effect of the metal ion on the normal cell process. A possible mode for toxicity increase may be considered in the light of the Tweedy chelation theory [28]. According to this theory, chelation considerably reduces the polarity of the metal ion because its positive charge is partly shared with the donor groups, and π electron delocalization over the entire chelate ring takes place. Such chelation could enhance the lipophilic character of the central metal atom, which subsequently favors its permeation through the lipid layers of cell membranes. Although there is a significant increase in the antibacterial activity of mixed-ligand complexes as compared to that of free ligands, the effect is less pronounced in comparison to that of the reference drug tetracycline.

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СИНТЕЗ, СПЕКТРАЛЬНЫЕ ХАРАКТЕРИСТИКИ И АНТИМИКРОБНЫЕ СВОЙСТВА КОМПЛЕКСОВ ПЕРЕХОДНЫХ МЕТАЛЛОВ ТРИДЕНТАТНЫМИ (ONS) И БИДЕНТАТНЫМИ (NN) ДОНОРНЫМИ ОСНОВАНИЯМИ ШИФФА

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Синтезирован ряд комплексов переходных металлов (Mn(II), Co(II), Ni(II), Cu(II), Zn(II) и Cd(II)) с салицилиден-*o*-аминотиофенолом и бис(бензилиден [или] ацетофенон)этилендиамином. Полученные соединения исследованы методами ИК и отражательно-абсорбционной спектроскопии, термогравиметрического анализа и магнитометрии. Исследованы антимикробные свойства комплексов в отношении штаммов *Salmonella typhimurium*, *Escherichia coli* и *Serratia marcescens*. Бактериостатический эффект комплексов выше по сравнению со свободными лигандами, хлоридами соответствующих металлов и ДМСО, но ниже чем у препарата сравнения тетрациклина.