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Metal Complexes: Preparation, Magnetic, Spectral, and Biocidal Studies of Some Mixed-Ligand Complexes with Schiff Bases Containing NO and NN Donor Atoms

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Metal Complexes: Preparation, Magnetic, Spectral, and Biocidal Studies of Some Mixed-Ligand Complexes with Schiff Bases Containing NO and NN Donor Atoms

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ABSTRACT

Metal complexes of the types $[M(SB^1)_2acphen]$ and $[M(SB^2)_2acphen]$ where M = Mn(II), Co(II), Ni(II), Cu(II), and Cd(II); $HSB^1 =$ 5-chlorosalicylidene-*p*-anisidine and $HSB^2 =$ 5-bromosalicylidene-*p*anisidine have been prepared and characterized. Magnetic susceptibility measurements, elemental analyses, electro-nic, and infrared spectral data suggest six-coordinated octahedral structures for these complexes. The mixed-ligand complexes show higher activity against *Saccharomyces cereuisae, Salmonella typhi, Fasarium oxysporum*,

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and *Lasiodiplodia theobromae* as compared to the ligand, metal salts, and control.

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Key Words: Metal complexes; Biocidal studies; Schiff bases; Electronic spectra.

INTRODUCTION

Schiff bases containing the -RC=N- group have gained importance because of their physiological and pharmacological activities.^[1] They constitute an interesting class of chelating agents capable of coordination with one or more metal ions giving mononuclear as well as polynuclear metal complexes, which serve as models for metalloproteins.^[2] In continuation of earlier work^[3] we report in the present communication the preparation, magnetic, spectral and biocidal studies of Mn(II), Co(II), Ni(II), Cu(II), and Cd(II) complexes with 5-chlorosalicylidene-*p*-anisidine or 5-bromosalicylidene-*p*anisidine as primary ligands and *bis*(acetophenone)ethylenediamine as a secondary ligand.

The structures of the ligands are shown in Fig. 1.



5-Chlorosalicylidene-*p*-anisidine (HSB¹) X = Cl
5-Bromosalicylidene-*p*-anisidine (HSB²) X = Br



Bis(acetophenone)ethylenediamine (acphen).

Figure 1. Structures of the ligands.



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EXPERIMENTAL

Preparation of the Schiff Bases

The preparation of 5-chlorosalicylidene-*p*-anisidine or 5-bromosalicylidene-*p*-anisidine was carried out by refluxing a solution 10 mmol of *p*-anisidene (1.23 g) in 100 mL ethanol with a solution of 10 mmol of 5-chlorosalicylaldehyde (1.56 g) or 5-bromosalicylaldehyde (2.00 g) in 100 mL ethanol for 2–3 hr. The reaction mixture was then allowed to cool to room temperature overnight. The yellow crystals were collected and dried in air.

The preparation of *bis*(acetophenone)ethylenediamine was carried out by refluxing a solution of 2 mmol of acetophenone (0.240 g) in 100 mL ethanol and a solution of 1 mmol of ethylenediamine (0.06 g) in 100 mL ethanol for 3–4 hr. The reaction mixture gave the Schiff base in the form of yellow crystals. The product was filtered and washed with ether and dried in air.

Preparation of the Complexes

bis(5-Chlorosalicylidene-*p*-anisidine)diaquoM(II) or *bis*(5-bromosalicylideneaniline)diaquaM(II), [M = Mn(II), Co(II), Ni(II), Cu(II), and Cd(II)] were synthesized by a published procedure.^[4] The preparation of the mixed-ligand complexes of *bis*(5-chlorosalicylidene-*p*-anisidine)acphenNi(II) or *bis*(5-bromosalicylidene-*p*-anisidine)acphenNi(II) was carried out by refluxing a solution of *bis*(5-chlorosalicylidene-*p*-anisidine)diaquaNi(II) (2 mmol, 1.23 g) or *bis*(5-bromosalicylidene-*p*-anisidine)diaquaNi(II) (2 mmol, 1.42 g) with dimethylformamide (100 mL) solution of *bis*(acetophenone)ethyl-enediamine (2 mmol, 0.52 g) for an hour. The solution was then concentrated to 15 mL, scratched and cooled in a refrigerator overnight. The obtained crystals were collected and recrystallized from chloroform. Finally, the crystals were dried in air. The complexes of Mn(II), Co(II), Cu(II), and Cd(II) were prepared similarly.

Analytical Procedure

Elemental analyses for carbon, hydrogen, and nitrogen were analyzed with a model 240 Perkin-Elmer elemental analyzer. Metal contents were determined by EDTA titration. The electronic spectra were recorded on a Beckman DK-2A spectrophotometer using MgO as a reference. The infrared spectra were measured on a FT-IR Nicolet 400 D spectrophotometer in KBr pellets. The melting points of the ligands and complexes were recorded in

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open capillaries in a capillary melting point apparatus (Tempo Instruments Co., Mumbai). The magnetic moments have been obtained in the solid state by a model 7304 vibrating sample magnetometer.

RESULTS AND DISCUSSION

The analytical data, colours, yields, and melting points of the ligands and complexes are presented in Table 1. The complexes were synthesized following the general procedures outlined by the following equations.

$$MCI_2 \cdot nH_2O + 2HSB^1 \xrightarrow{H_2O} [M(SB^1)_2(H_2O)_2] + 2HCl$$
(1)

$$[M(SB^{1})_{2}(H_{2}O)_{2}] + acphen \xrightarrow{EtOH, Reflux} [M(SB^{1})_{2}acphen] + 2H_{2}O \quad (2)$$

$$MCI_2 \cdot nH_2O + 2HSB^2 \xrightarrow{H_2O} [M(SB^2)_2(H_2O)_2] + 2HCl$$
(3)

$$[M(SB2)2(H2O)2] + acphen \xrightarrow{EtOH, Reflux} [M(SB2)2acphen] + 2H2O (4)$$

Magnetic Measurements

The magnetic moment data are presented in Table 1. In the present case the magnetic moment values of $[Mn(SB^1)_2acphen]$ and $[Mn(SB^2)_2acphen]$ are 6.1 and 5.9 B.M., respectively, which is within the limits of the spin-free value for five unpaired electrons, indicating that the complexes are high-spin d⁵ system with an octahedral geometry.^[5] The magnetic moment data of $[Co(SB^1)_2acphen]$ and $[Co(SB^2)_2acphen]$ are 4.71 and 4.60 B.M., respectively,^[6] suggesting an octahedral geometry. The magnetic moment data of $[Ni(SB^1)_2acphen]$ and $[Ni(SB^2)_2acphen]$ are 2.88 and 2.81 B.M., respectively, which are in the usual range expected for similar hexa-coordinated Ni(II) ions.^[7] The magnetic moment data of $[Cu(SB^2)_2acphen]$ are 1.75 and 1.76 B.M., respectively, which are very close to the spin-only value (1.73 B.M.) expected for one unpaired electron, which offers the possibility of an octahedral geometry.^[8] The magnetic moment determinations show that the Cd(II) complexes are diamagnetic.

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		Table 1.	Analytical da	tta of the ligar	nds and compl	exes. ^a			
				% Found	ł (calcd.)				
Compounds empirical formula	Formula weight	Colour	С	Н	Ν	Metal	m.p. (°C)	$\mu_{\rm eff}$ (B.M.)	Yield % (g)
$HSB^{1}, C_{14}H_{12}CINO_{2}$	261.00	Yellow	64.32 (64.36)	4.55 (4.59)	5.36 (5.32)		250	I	75.0 (2.18)
HSB ² , C ₁₄ H ₁₂ BrNO ₂	305.90	Yellow	54.90 (54.91)	3.90 (3.92)	4.52 (4.57)		255		70.0 (2.34)
acphen, $C_{18}H_{20}N_2$	264.00	Yellow	81.79 (81.81)	7.60 (7.67)	10.54 (10.60)		260		75.0 (1.35)
[Mn(SB ¹) ₂ acphen]	838.94	Yellowish	65.80 (65.79)	4.99 (5.00)	6.66 (6.67)	6.58 (6.54)	278	6.1	58.0 (0.23)
$C_{46}H_{42}Cl_2MnN_4O_4$		brown							
[Co(SB ¹) ₂ acphen]	842.94	Light	65.42 (65.48)	4.93 (4.98)	6.60 (6.64)	6.95 (6.99)	285	4.71	78.0 (088)
$C_{46}H_{42}Cl_2CoN_4O_4$		brown							
[Ni(SB ¹) ₂ acphen]	842.71	Green	65.51 (65.50)	4.95 (4.98)	6.62 (6.64)	6.90 (6.96)	285	2.88	70.0 (0.80)
$C_{46}H_{42}Cl_2NiN_4O_4$									
[Cu(SB ¹) ₂ acphen]	847.55	Greenish	65.10(65.12)	4.96 (4.95)	6.60 (6.60)	7.45 (7.49)	285	1.75	68.0 (0.77)
$C_{46}H_{42}Cl_2CuN_4O_4$		brown							
[Cd(SB ¹) ₂ acphen]	896.40	Yellow	61.55 (61.57)	4.69(4.68)	6.20 (6.24)	12.50 (12.53)	270		69.0(0.81)
$C_{46}H_{42}Cl_2CdN_4O_4$									
[Mn(SB ²) ₂ acphen]	928.74	Yellowish	59.40 (59.43)	4.50 (4.52)	6.00 (6.02)	5.90 (5.91)	280	5.9	70.0 (0.86)
$\mathrm{C}_{46}\mathrm{H}_{42}\mathrm{Br}_{2}\mathrm{MnN}_{4}\mathrm{O}_{4}$		brown							
[Co(SB ²) ₂ acphen]	932.74	Light	59.15 (59.18)	4.45 (4.50)	5.99 (6.00)	6.38 (6.31)	280	4.60	70.0 (0.85)
$\mathrm{C}_{46}\mathrm{H}_{42}\mathrm{Br}_{2}\mathrm{CoN}_{4}\mathrm{O}_{4}$		brown							
[Ni(SB ²) ₂ acphen]	932.51	Green	59.22 (59.19)	4.55 (4.50)	5.99 (5.98)	6.25 (6.29)	279	2.81	67.0 (0.78)
$\mathrm{C}_{46}\mathrm{H}_{42}\mathrm{Br}_{2}\mathrm{NiN}_{4}\mathrm{O}_{4}$									
[Cu(SB ²) ₂ acphen]	937.35	Greenish	58.85 (58.87)	4.46 (4.47)	5.95 (5.97)	6.75 (6.77)	280	1.80	78.0 (0.96)
$C_{46}H_{42}Br_2CuN_4O_4$		brown							
[Cd(SB ²) ₂ acphen]	986.20	Yellow	55.95 (55.97)	4.20 (4.25)	6.00 (6.02)	11.35 (11.39)	280		60.0 (0.76)
$C_{46}H_{42}Br_2CdN_4O_4$									
^a HSB ¹ , 5-chlorosalicyli	dene-p-anis	idene; HSB ² ,	5-bromosalicyl	lidene-p-anisio	lene.				

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Electronic Spectra

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The electronic spectra of the complexes were recorded in the solid phase at room temperature. The electronic spectra of the Mn(II) complexes^[9] gave three bands at \sim 15,000, \sim 19,000 and \sim 25,500 cm⁻¹. These bands may be assigned to the ${}^{6}A_{1g} \rightarrow {}^{4}T_{1g}({}^{4}G)(\nu_{1}), {}^{6}A_{1g} \rightarrow {}^{4}T_{2g}({}^{4}G)(\nu_{2})$ and ${}^{6}A_{1g} \rightarrow {}^{4}A_{1g}, {}^{4}E_{g}({}^{4}G)(\nu_{3})$ transitions in order of increasing energy. The electronic spectra of the Co(II) complexes exhibit three bands at ~9,300, ~18,000, and ~19,000 cm⁻¹, which may be reasonably assigned^[10] to the ${}^{4}T_{1g}(F) \rightarrow {}^{4}T_{2g}(F) (\nu_1), {}^{4}T_{1g}(F) \rightarrow {}^{4}A_{2g}(\nu)$, and ${}^{4}T_{1g}(F) \rightarrow {}^{4}T_{1g}(P) (\nu_3)$ transitions, consistent with an octahedral geometry around the metal ion. The electronic spectra of the Ni(II) complexes gave three bands at \sim 10,500, \sim 18,000 and ~24,000 cm⁻¹, which may be ascribed^[11] to the ${}^{3}A_{2g}(F) \rightarrow {}^{3}T_{2g}(F)(\nu_{1}), {}^{3}A_{2g}(F)$ $(F) \rightarrow {}^{3}T_{1g}(F)(\nu_{2}) \text{ and } {}^{3}A_{2g}(F) \rightarrow {}^{3}T_{1g}(P)(\nu_{3}) \text{ transitions, respectively, corre$ sponding to an octahedral geometry of the Ni(II) ion. The electronic spectra of the Cu(II) complexes exhibit one band at ~15,000 cm⁻¹ assignable to the ${}^{2}E_{p}$ - $\rightarrow {}^{2}T_{2g}(F)$ transition, corresponding^[12] to an octahedral geometry of the Cu(II) ion. The value of the ligand field splitting energy (D_q) , Racah interelectronic repulsion parameter (B), nephelauxetic ratio (β), and ratio v_2/v_1 for the Ni(II) and Co(II) complexes are presented in Table 2.

Infrared Spectra

A comparision of the characteristic IR absorption bands of the ligands with those of the corresponding Mn(II), Co(II), Ni(II), Cu(II), and Cd(II) complexes reveals the following important features. In the infrared spectra of the 5-chlorosalicylidene-p-anisidine and 5-bromosalicylidene-p-anisidine ligands, a relatively sharp band appearing at $\sim 2700 \,\mathrm{cm}^{-1}$ may be assigned to the v(OH) vibrations.^[13] The absence of this band in the complexes indicates the deprotonation of the phenolic groups and coordination of the oxygen atoms to the metal ion. In the free ligand, a strong band at $\sim 1525 \text{ cm}^{-1}$ due to ν (C–O) (phenolic) shifts to higher energy by 5–10 cm⁻¹ in the complexes indicating the coordination of the phenolic oxygen atom to the metal ion.^[14] This shift of the band to higher energy is expected due to the maintenance of ring currents arising from the electron delocalization in the chelate ring. The ν (C = N) frequency of the *bis*(acetophenone) ethylenediamine, 5-chlorosalicylidene-p-anisidine, and 5-bromosalicylidene-p-ani- sidine ligands occur at $\sim 1640 \,\mathrm{cm}^{-1}$. This band shifts to lower energy by $10-20 \,\mathrm{cm}^{-1}$ in the mixed-ligand complexes indicating coordination through the azomethine nitrogen.^[15] The complexes of 5-chlorosalicylidene-p-anisidine exhibit a

	Tabl	e 2. Electroni	ic spectral data	of the Ni(II) ar	nd Co(II) comp	olexes. ^a		
	Obse	erved bands (cn	\mathfrak{n}^{-1})					
Complexes	ν_1	ν_2	ν_3	$ u_2/ u_1 $	В	β	eta°	$D_{ m q}$
[Co(SB ¹) ₂ acphen]	9,300	18,200	19,000	2.00	739	0.760	23.95	1,020
$[Ni(SB^{1})_{2}acphen]$	10,500	18,000	24,000	1.66	676	0.600	34.50	1,050
[Co(SB ²) ₂ acphen]	9,250	18,181	19,000	2.00	739	0.770	25.94	1,020
[Ni(SB ²) ₂ acphen]	10,400	17,500	24,000	1.70	716	0.698	30.50	1,040
^a HSB ¹ , 5-chlorosalicyl	idene- <i>p</i> -aniside	ene; HSB ² , 5-b	romosalicylide	ne-p-anisidene.				

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band at ~725 cm⁻¹ due to ν (C–Cl).^[16] The complexes of 5-bromosalicylidene*p*-anisidine exhibit band at ~690 cm⁻¹ due to ν (C–Br).^[17] The frequencies in the range 2815–2830 cm⁻¹ and 1370–1385 cm⁻¹ are due to ν (C–OCH₃).^[18] and ν (C–CH₃),^[19] respectively. The frequencies in the range 450–460 cm⁻¹ and 410–420 cm⁻¹ are attributed to ν (M–N).^[20] and ν (M–O),^[21] respectively. The suggested structures of the complexes are shown in Fig. 2.

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Antimicrobial Activity

The antimicrobial activity of the ligand, the metal salts and their corresponding complexes were tested against *Salmonella typhi* (bacteria), *Saccharomyces cereuisae* (yeast), and two fungal species *Lasiodiplodia theobromae* and *Fusarium oxysporum*. The antimicrobial activities of the compounds were tested against bacteria and yeast using the "Agar Diffusion Method".^[22] The fungal activity of the complexes has been screened against *Lasiodiplodia theobromae* and *Fusarium oxysporum* using the "Potato Dextrose Agar Method".^[23] All of the compounds have significant antimicrobial activity at $5 \times 10^{-4} \text{ g mL}^{-1}$ against bacteria, yeast, and fungi. The compounds are soluble in dimethylformamide. Therefore, the growth was compared with dimethylformamide as the control and is expressed as percentage inhibition



 $[M(SB^{1})_{2}acphen](X = Cl) or [M(SB^{2})_{2}acphen](X = Br)$

Figure 2. Suggested structure of the complexes.

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Table 3. Antimicrobial activities of the complexes.^a

		% Inhil	oition	
	Destario	Vaast	Fun	gi
Compounds	Salmonella typhi.	Saccharomyces cereuisae	Lasiodiplodia theobromae	Fusarium oxysporum
$MnCl_2 \cdot 4H_2O$	22.22	21.05	20.00	15.30
$CoCl_2 \cdot 6H_2O$	15.16	10.52	06.60	07.70
$NiCl_2 \cdot 6H_2O$	11.11	8.26	10.30	10.30
$CuCl_2 \cdot 2H_2O$	22.20	15.78	20.00	15.30
$CdCl_2 \cdot 2H_2O$	16.66	08.26	06.60	07.70
HSB^1	22.22	15.78	06.60	23.00
HSB^2	27.77	26.31	13.30	37.00
acphen	38.88	10.50	26.00	23.00
[Mn(SB ¹) ₂ acphen]	55.00	57.89	33.30	46.00
[Co(SB ¹) ₂ acphen]	50.00	68.42	33.30	53.00
[Ni(SB ¹) ₂ acphen]	55.00	57.89	33.30	46.00
[Cu(SB ¹) ₂ acphen]	61.11	63.15	40.00	46.00
[Cd(SB ¹) ₂ acphen]	44.40	63.15	40.00	53.00
[Mn(SB ²) ₂ acphen]	61.11	57.89	36.60	46.00
[Co(SB ²) ₂ acphen]	50.00	47.36	46.60	47.00
[Ni(SB ²) ₂ acphen]	61.11	52.63	47.00	50.00
[Cu(SB ²) ₂ acphen]	50.00	52.63	45.00	53.00
[Cd(SB ²) ₂ acphen]	50.00	52.89	40.00	45.00

^aHSB¹, 5-chlorosalicylidene-*p*-anisidene; HSB², 5-bromosalicylidene-*p*-anisidene.

vs. controls. The percentage inhibition of various compounds are recorded in Table 3. The data represents the values of the five replicates. From the data it is observed that the complexes show higher activity against bacteria, yeast, and fungi as compared to the control (blank), the ligands and metal salts. The results suggest that the changes in structure due to the coordination in the complexes decreases the growth of microorganisms relative to the metal ion and ligands. Hence, complexation increases the antimicrobial activity due to presence of two halogen and four azomethine (C==N) groups. The complexes show higher activity against *Salmonella typhi* and *Fusarium oxysporum* as compared to *Saccharomyces cereuisae* and *Lasiodiplodia thebromae*. The complexes are more active against *Fusarium oxysporum* as compared to *Lasiodiplodia thebromae*. The patterns of activity are complex and no clear trend can be ascertained.



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