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# Synthesis and Reactivity in Inorganic, Metal-Organic, and Nano-Metal Chemistry

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# New Cobalt and Zinc Complexes with Schiff Base Ligands: Synthesis, Structures, and Biochemical Properties

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Two similar Schiff base ligands 2-[(2-isopropylaminoethylimino)methyl]-5-methoxyphenol (HL<sup>a</sup>) and 2-ethoxy-6-[(2-isopropylaminoethylimino)methyl]phenol (HL<sup>b</sup>) reacting with cobalt perchlorate and zinc iodide, respectively, in methanol gave the complexes  $[CoL^a_2] \cdot ClO_4$  (1) and  $[Zn(HL^b)_2I_2(\mu-OH)] \cdot I \cdot MeOH$  (2). Both complexes were characterized by elemental analyses and IR spectroscopic method in the solid state. Single-crystal X-ray analysis was performed, which reveals that (1) is a mononuclear cobalt complex, and (2) is a dinuclear zinc complex. The Co atom in (1) is in an octahedral coordination. The Zn atoms in (2) are in tetrahedral coordination. Antibacterial activities of the Schiff base ligands and the complexes have been studied on the strains *B. subtilis, E. coli*, and *S. aureus*.

Keywords: Biochemical property, cobalt complex, crystal structure, Schiff base, zinc complex

#### Introduction

Experimental

Schiff bases in general have been shown to be biological active. A great deal of Schiff bases were reported to possess antibacterial, antifungal and antitumor activities.<sup>[1-3]</sup> The effect of presence of various substituents in the phenyl rings of aromatic Schiff bases on their antibacterial activity has been reported.<sup>[4]</sup> Due to their multiple implications, the transition metal complexes with Schiff bases, as ligands, are of paramount scientific interest. It has been found that in general the complexation of Schiff bases with most transition metal atoms influences their antibacterial activities.<sup>[5-7]</sup> In recent years, cobalt and zinc complexes have been extensively used in medicine.<sup>[8–10]</sup> With this view and in continuation of work on the study of Schiff bases and their metal complexes, herein we report two new cobalt and zinc complexes,  $[CoL^{a}_{2}] \cdot ClO_{4}$  (1) and  $[Zn(HL^{b})_{2}I_{2}(\mu-OH)] \cdot I \cdot MeOH$  (2), where L<sup>a</sup> is the monoanionic form of 2-[(2-isopropylaminoethylimino)methyl]-5-methoxyphenol (HL<sup>a</sup>) and HL<sub>b</sub> is the zwitterionic form of 2-ethoxy-6-[(2-isopropylaminoethylimino)methyl]phenol (HL<sup>b</sup>). The antibacterial activity of the Schiff bases and their complexes are reported against B. subtilis, E. coli, and S. aureus.

#### Materials and Methods

4-Methoxysalicylaldehyde, 3-ethoxysalicylaldehyde, and N-isopropylethane-1,2-diamine were purchased from Merck and Fluka, and used as received. The Schiff base ligands were prepared in over 90% yields by condensation reactions of N-isopropylethane-1,2-diamine with 4-methoxysalicylaldehyde and 3-ethoxysalicylaldehyde, respectively, in methanolic media, according to the literature method.<sup>[11]</sup> Cobalt perchlorate was prepared by the reaction of cobalt carbonate with perchloric acid in aqueous solution. All other chemicals and solvents used in this work were of analytical grade available commercially and used without further purification. Elemental analyses (carbon, hydrogen, and nitrogen) of the compounds were obtained from a Carlo ERBA Model EA 1108 analyzer. Infrared spectra were collected by using KBr pellets on a Jasco-5300 FT-IR spectrophotometer. Solution electrical conductivity was measured with a DDS-11A conductivity meter.

#### Synthesis of $[CoL_2^a] \cdot ClO_4$ (1)

Cobalt perchlorate heptahydrate (0.01 mmol, 3.83 g) dissolved in methanol (30 mL) was added dropwise to a stirred methanolic solution (30 mL) of HL<sup>a</sup> (0.01 mol, 2.36 g). The mixture was gently refluxed for 2 h, then most of the solvent was evaporated by distillation. After cooling, the resulting brown solid was filtered off, washed with cold methanol, and dried in a vacuum containing anhydrous CaCl<sub>2</sub>. Yield: 2.32 g (74%). Anal. Calcd. for C<sub>26</sub>H<sub>38</sub>ClCoN<sub>4</sub>O<sub>8</sub> (%): C, 49.65; H,

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6.09; N, 8.91. Found: C, 49.53; H, 6.17; N, 8.82. Brown block-like single crystals of the complex, suitable for single-crystal X-ray diffraction, were obtained by slow evaporation of a methanol solution containing the complex.

### Synthesis of $[Zn(HL^b)_2I_2(\mu-OH)] \cdot I \cdot MeOH$ (2)

Zinc iodide (0.01 mmol, 3.19 g) dissolved in methanol (30 mL) was added dropwise to a stirred methanolic solution (30 mL) of HL<sup>b</sup> (0.01 mol, 2.50 g). The mixture was gently refluxed for 2 h, then most of the solvent was evaporated by distillation. After cooling, the resulting colorless solid was filtered off, washed with cold methanol, and dried in a vacuum containing anhydrous CaCl<sub>2</sub>. Yield: 2.77 g (53%). Anal. Calcd. for  $C_{57}H_{88}I_6N_8O_{11}Zn_4$  (%): C, 32.85; H, 4.26; N, 5.38. Found: C, 32.70; H, 4.37; N, 5.45. Colorless needle-like single crystals of the complex, suitable for single crystal X-ray diffraction, were obtained by slow evaporation of a methanol solution containing the complex.

#### X-Ray Crystallography

Suitable X-ray quality crystals of the complexes (1) and (2) were pick up under a microscope and investigated in a diffraction experiment at 298(2) K on a Bruker Apex II diffractometer with monochromated Mo K $\alpha$  radiation ( $\lambda = 0.71073$  Å) obtained from a graded multilayer X-ray optics. The structures were solved by direct methods with SHELXS-97,<sup>[12]</sup> and refined with full-matrix least-squares techniques on  $F^2$  with SHELXL-97.<sup>[12]</sup> The C- and O-bonded hydrogen

atoms were calculated in an idealized geometry, riding on their parent atoms, with distances restrained to 0.93-0.97 Å for C—H and 0.86-0.91 Å for N–H. The N4-C24-C25-C26 moiety in complex (2) is disordered with occupancies of 0.55 (2) and 0.45(2). The crystal data and refinement parameters are listed in Table 1. Selected bond lengths and angles are listed in Table 2.

#### Antibacterial Assay

The antibacterial activities were tested against B. subtilis, E. *coli*, and *S. aureus* using Mueller-Hinton medium. The MICs (minimum inhibitory concentrations) of the test compounds were determined by a colorimetric method using the dye MTT [3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyl tetrazolium bromide]. A stock solution of the synthesized compound (50  $\mu$ g mL<sup>-1</sup>) in DMSO was prepared and quantities of the test compounds were incorporated in specified quantity of sterilized liquid Mueller-Hinton medium. A specified quantity of the medium containing the compound was poured into microtitration plates. A suspension of the microorganism was prepared to contain approximately  $10^5$  cfu mL<sup>-1</sup> and applied to microtitration plates with serially diluted compounds in DMSO to be tested and incubated at 37°C for 24 h. After the MICs were visually determined on each of the microtitration plates, 50  $\mu$ L of PBS (phosphate buffered saline 0.01 mol  $L^{-1}$ , pH 7.4: Na<sub>2</sub>HPO<sub>4</sub>·12H<sub>2</sub>O 2.9 g, KH<sub>2</sub>PO<sub>4</sub> 0.2 g, NaCl 8.0 g, KCl 0.2 g, distilled water 1000 mL) containing 2 mg of MTT/mL was added to each well. Incubation was continued at room temperature for

**TABLE 1.** Crystallographic data for the complexes.

	(1)	(2)	
Chemical formula	C <sub>26</sub> H <sub>38</sub> ClCoN <sub>4</sub> O <sub>8</sub>	C <sub>57</sub> H <sub>88</sub> I <sub>6</sub> N <sub>8</sub> O <sub>11</sub> Zn <sub>4</sub>	
Formula weight	628.98	2084.23	
Crystal system	Orthorhombic	Monoclinic	
Space group	$Pna2_1$	C2/c	
Unit cell dimensions			
a (Å)	12.350(2)	29.962(1)	
$b(\mathbf{A})$	13.243(2)	14.5860(5)	
c (Å)	18.087(2)	19.7055(9)	
$\alpha$ (°)	90	90	
$\beta(\circ)$	90	118.024(3)	
$\gamma$ (°)	90	90	
$V(Å^3)$	2958.1(7)	7602.1(5)	
Z	4	4	
$\rho (\mathrm{g  cm^{-3}})$	1.412	1.821	
$\mu (\mathrm{mm}^{-1})$	0.723	3.738	
$T_{\min}, T_{\max}$	0.8869, 0.8993	0.5369, 0.6040	
Reflections collected	12852	21831	
Reflections unique	5110	7842	
Reflections observed $[I > 2\sigma(I)]$	4149	5450	
Parameters	367	425	
Restraints	82	14	
$R_1, wR_2 [I > 2\sigma(I)]$	0.0989, 0.2483	0.0375, 0.0777	
$R_1, wR_2$ (all data)	0.1152, 0.2656	0.0638, 0.0885	
Goodness-of-fit on $F^2$	1.029	1.052	



**FIG. 1.** Structure of complex (1) with the atom labeling scheme. All non-hydrogen atoms are represented at 30% probability thermal ellipsoids. Hydrogen atoms are omitted for clarity.

4–5 h. The content of each well was removed and 100  $\mu$ L of isopropanol containing 5% 1 mol L<sup>-1</sup> HCl was added to extract the dye. After 12 h of incubation at room temperature, the optical density (OD) was measured with a microplate reader at 550 nm. The antibiotics kanamycin and penicillin were used as standard drugs. The observed MIC values are given in Table 3.

#### **Results and Discussion**

#### Synthesis and Characterization

Reaction of cobalt perchlorate with HL<sup>a</sup> and zinc iodide with HL<sup>b</sup> in methanol, respectively, under aerobic conditions afforded mononuclear cobalt complex (1) and dinuclear zinc complex (2). Both complexes were obtained as single crystals, stable in air and soluble in polar organic solvents such as ethanol, methanol, DMF, and DMSO. Elemental analyses of the complexes are consistent with the general molecular formulae proposed by single-crystal X-ray determination. The molar conductivity of the complexes in absolute methanolic solution confirms the 1:1 electrically nature.<sup>[13]</sup>

#### **IR Spectroscopic Characteristics**

Comparison of the IR spectra of the complexes with those of the free Schiff bases gives information about the coordination. The IR spectra of the ligands exhibit broad bands in the range 3300–3500 cm<sup>-1</sup> due to phenolic (O–H) stretching vibrations. The weak and sharp bands in the range 3150– 3270 cm<sup>-1</sup> for the ligands and the complexes are assignable to the N–H stretching. The complexes display strong peaks at about 1610 cm<sup>-1</sup> due to azomethine (C=N) stretching.<sup>[14]</sup> The azomethine  $\nu$ (C = N) bands in the complexes are shifted by about 15–20 cm<sup>-1</sup> to lower wave numbers compared to the free Schiff base ligands. For the spectra of the Schiff base ligands, the strong bands at about 1260 cm<sup>-1</sup> are due to

TABLE 2. Selected bond lengths (Å) and angles (°) for the complexes.

(1)			
Bond lengths (Å)			
Co1-O1	1.883(8)	Co1-O3	1.877(11)
Col-N1	1.898(8)	Co1-N2	2.099(11)
Co1-N3	1.903(9)	Co1-N4	1.990(11)
Bond angles (°)			
O3-Co1-O1	87.9(3)	O3-Co1-N1	92.9(3)
01-Co1-N1	87.8(4)	O3-Co1-N3	87.2(4)
O1-Co1-N3	94.7(3)	N1-Co1-N3	177.5(4)
O3-Co1-N4	89.3(5)	O1-Co1-N4	177.0(5)
N1-Co1-N4	93.3(4)	N3-Co1-N4	84.2(4)
O3-Co1-N2	177.1(4)	O1-Co1-N2	90.0(4)
N1-Co1-N2	85.1(4)	N3-Co1-N2	94.9(4)
N4-Co1-N2	92.9(3)		
(2)			
Bond lengths (Å)			
Zn1-O1	1.949(3)	Zn1-O5	1.934(3)
Zn1-N1	2.011(4)	Zn1-I2	2.5313(7)
Zn2-O3	1.950(3)	Zn2-O5	1.926(3)
Zn2-N3	1.987(4)	Zn2-I1	2.5717(6)
Bond angles (°)			
O5-Zn1-O1	100.36(13)	O5-Zn1-N1	114.04(15)
O1-Zn1-N1	95.86(14)	O5-Zn1-I2	115.35(9)
O1-Zn1-I2	121.67(10)	N1-Zn1-I2	108.19(11)
O5-Zn2-O3	104.64(13)	O5-Zn2-N3	112.06(15)
O3-Zn2-N3	96.56(14)	O5-Zn2-I1	110.50(9)
O3-Zn2-I1	122.01(9)	N3-Zn2-I1	110.31(11)

 $\nu$ C—O(phenolic).<sup>[15]</sup> While for the complexes, the C—O stretching bands are observed at about 1240 cm<sup>-1</sup>. These spectra assignments show that the Schiff bases are coordinated to the metal atoms through azomethine nitrogen and phenolic oxygen. Conclusive evidence of the bonding is also shown by the presence of new bands in the spectra of the complexes at low wave numbers 430–570 cm<sup>-1</sup>, which can be attributed to M-O and M-N stretching vibrations. A strong  $\nu$ (ClO<sub>4</sub><sup>-</sup>) at 1087 cm<sup>-1</sup> and a medium absorption at 623 cm<sup>-1</sup> for complex (1) indicate the presence of uncoordinated ClO<sub>4</sub><sup>-</sup>.<sup>[16]</sup>

#### Description of the Crystal Structure of (1)

Figure 1 gives the ORTEP diagram with atomic labeling scheme of complex (1). The asymmetric unit of the compound contains a mononuclear cobalt complex cation and a

**TABLE 3.** MIC values ( $\mu g m L^{-1}$ ) of the tested compounds.

	B. subtilis	E. coli	S. aureus
HL <sup>a</sup>	>100	25	>100
HL <sup>b</sup>	>100	25	>100
(1)	1.56	6.25	6.25
(2)	6.25	12.5	25
Penicillin	0.78	>100	3.13
Kanamycin	0.39	6.25	1.56



FIG. 2. Hydrogen bond linked structure of complex (1).

perchlorate anion. In the complex cation, two of the monoanionic tridentate Schiff base ligands wrap the Co center in the *cis* mode with coordination of two oxygens and four nitrogens in a distorted octahedral coordination. The two phenolic hydroxyls are deprotonated on complexation. The azomethine (C=N) bond lengths of 1.266(12) and 1.282(14) Å are within the general values of C=N double bonds. Most *cis* and *trans* angles at the metal center are in the range 84.2(4)– 94.9(4)° and 177.0(5)–177.5(4)°, respectively. The *trans* angles differ by about 3° from the ideal ones because of ligand restraints imposed by the adjacent five- and six-membered chelate rings. The average Co–O/N distances in the complex compare well with those reported for similar cobalt complexes with Schiff bases.<sup>[15,17,18]</sup>

The crystal packing of the complex is shown in Figure 2. The perchlorate anions are linked to the complex cations through intermolecular N–H...O hydrogen bonds.

FIG. 4. Hydrogen bond linked structure of complex (2).

#### Description of the Crystal Structure of (2)

Figure 3 gives the ORTEP diagram with atomic labeling scheme of complex (2). Selected bond lengths and angles are given in Table 2. The asymmetric unit of the compound contains a hydroxy bridged dinuclear zinc complex cation, an iodide anion, and a methanol molecule. The zinc atom in the complex are tetrahedrally coordinated by one phenolic oxygen atom, one imine nitrogen atom from the Schiff base ligand, one iodide atom, and one hydroxy oxygen atom. The coordination about the zinc atoms is distorted due to the restricting bite angles of the chelating ligands. The N1-Zn1-O1 and N3-Zn2-O3 angles are 95.86(14) and 96.56(14)°, being in the same range of 90–98° found for similar zinc com-plexes with Schiff base ligands.<sup>[19,20]</sup> The I1-Zn2-O5 and I2-Zn1-O5 angles are 110.50(9) and 115.35(9)°, respectively. The deviation from regular tetrahedral angle of 109.5° is due to the ligand straints imposed by the six-membered chelate rings. The average Zn-N, Zn-O, and Zn-I bond lengths agree



**FIG. 3.** Structure of complex (2) with the atom labeling scheme. All non-hydrogen atoms are represented at 30% probability thermal ellipsoids. Hydrogen atoms are omitted for clarity.



well with corresponding bond lengths in other complexes with tetrahedrally coordinated zinc atoms.<sup>[21,22]</sup>

The crystal packing of the complex is shown in Figure 4. The dinuclear zinc complex cations are linked by iodide anions through intermolecular  $N-H\cdots I$  hydrogen bonds, to form 1D chains running along the *a*-axis.

#### Antibacterial Activities

The Schiff base ligands  $HL^a$  and  $HL^b$  have also the same activities against *E. coli*, and no activity again from *B. subtilis* and *S. aureus*. The cobalt and zinc complexes have from weak to medium activities against the three strains. In general, the complexes have stronger antibacterial activities than the free Schiff base ligands. It is obvious that complex (1) has stronger activities against the bacteria than complex (2). However, when compared with Penicillin and Kanamycin, the activities of the Schiff bases and the complexes are far weaker. Detailed comparison among the compounds can simply conclude that the substitute groups and their positions of the Schiff base ligands are not crucial for the antibacterial activity. Yet, the combination of metal atoms can enhance the antibacterial activities.

#### Conclusions

New cobalt and zinc complexes were synthesized and characterized with the help of microanalytical, IR spectroscopic, and single-crystal X-ray crystallographic measurements. The crystal structures of the complexes reveal that the Co atom is in a distorted octahedral coordination sphere and the Zn atoms are in tetrahedral coordination spheres. The complexes have from weak to medium antibacterial activities on the strains *B. subtilis, E. coli*, and *S. aureus*.

#### **Supplemental Material**

CCDC 934999 (1) and 935000 (2) contain the supplementary crystallographic data for the complex. These data can be obtained free of charge *via* http://www.ccdc.cam.ac.uk/ conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam. ac.uk.

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