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

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## Synthesis, Crystal Structures, and Antibacterial Activity of Copper(II) and Cobalt(III) Complexes Derived from 2-{1-[2-(2-Hydroxyethylamino)ethylimino]ethyl}phenol

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## Synthesis, Crystal Structures, and Antibacterial Activity of Copper(II) and Cobalt(III) Complexes Derived from 2-{1-[2-(2-Hydroxyethylamino)ethylimino]ethyl}phenol

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A new copper(II) complex [Cu(HEP)(N<sub>3</sub>)] (1), and a new cobalt(III) complex [Co(HEP)(AE)]·Cl·H<sub>2</sub>O (2), where HEP is 2-{1-[2-(2-hydroxyethylamino)ethylimino]ethyl}phenolate, AE is 2-(2-aminoethylamino)ethanol, were prepared and characterized by elemental analysis, IR spectra, and single crystal X-ray diffraction. Complex (1) crystallizes in the monoclinic space group  $P2_1/c$  with unit cell dimensions a = 9.969(3) Å, b = 19.218(3) Å, c = 7.272(2)Å,  $\beta = 108.788(3)^\circ$ , V = 1319.0(6) Å<sup>3</sup>, Z = 4,  $R_1 = 0.0424$  and  $wR_2 = 0.1116$ . Complex (2) crystallizes in the triclinic space group P-1 with unit cell dimensions a = 7.205(2) Å, b = 9.976(3) Å, c =16.737(5) Å,  $\alpha = 98.522(4)^\circ$ ,  $\beta = 102.076(4)^\circ$ ,  $\beta = 96.218(4)^\circ$ , V = 1151.4(6) Å<sup>3</sup>, Z = 2,  $R_1 = 0.0614$  and  $wR_2 = 0.1308$ . The Cu atom in (1) is four-coordinate in a square planar geometry. The Co atom in (2) is six-coordinate in an octahedral geometry. The in vitro antibacterial activity of the ligands and the complexes was tested against the bacteria Staphylococcus aureus, Bacillus subtilis, Klebsiella pneumonia, Pseudomonas aeruginosa and Escherichia coli.

Keywords antibacterial activity, cobalt, copper, crystal structure, Schiff base, synthesis

#### 1. INTRODUCTION

Schiff bases derived from the condensation of aldehydes with organic amines are a kind of versatile ligands in coordination chemistry due to their easy synthesis and wide applications.<sup>[1-4]</sup> Considerable attention has been focused on the biological properties of Schiff bases and their metal complexes.<sup>[5-7]</sup> Recent research indicates that in the presence of metal ions, the phenolic-containing compounds are believed to damage the cytoplasmic membrane, and show potential antimicrobial activity.<sup>[8–9]</sup> In this paper, a new copper(II) complex [Cu(HEP)(N<sub>3</sub>)] (1), and a new cobalt(III) complex [Co(HEP)(AE)]·Cl·H<sub>2</sub>O (2), where HEP is 2-{1-[2-(2-hydroxyethylamino)ethylimino]ethyl}phenolate (Scheme 1), AE is 2-(2-aminoethylamino)ethanol, were prepared and characterized. The in vitro antibacterial activity of the ligands and the complexes was evaluated. To our knowledge, only one copper(II) complex derived from the Schiff base ligand has been reported.<sup>[10]</sup>

#### 2. EXPERIMENTAL

#### 2.1. Materials and Measurements

All chemicals were available commercially as AR grade and were used without further purification. C, H, and N elemental analyses were performed on a Vario-EL-III analyzer. The IR spectra were recorded on a Perkin Elmer 2000 spectrophotometer with KBr pellets in the region 4000–400 cm<sup>-1</sup>.

Caution: Sodium azide and copper(II) perchlorate are potentially explosive, only a small quantity should be used and handled with great care.

#### 2.2. Synthesis of the Schiff Base Ligand

1-(2-Hydroxyphenyl)ethanone (1.0 mmol, 136.2 mg) and 2-(2-aminoethylamino)ethanol (1.0 mmol, 104.2 mg) were mixed in 50 mL methanol and then stirred and refluxed for 30 min. The solvent was removed by distillation to give yellow product. Yield: 93%. Analysis calculated for  $C_{12}H_{18}N_2O_2$ : C, 64.8; H, 8.2; N, 12.6%; found: C, 64.6; H, 8.2; N, 12.7%.

#### 2.3. Synthesis of $[Cu(HEP)(N_3)]$ (1)

A methanol solution (10 mL) of copper(II) perchlorate (0.1 mmol, 37.0 mg) was added with stirring to a methanol solution (10 mL) of the Schiff base (0.1 mmol, 22.2 mg), 2-(2-aminoethylamino)ethanol (0.1 mmol, 10.4 mg), and sodium azide (0.1 mmol, 6.5 mg). The mixture was stirred at ambient conditions for 30 min to give a blue solution, which was

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SCH. 1. The Schiff base ligand.

allowed to evaporate slowly in air for a week, yielding blue single crystals, which were collected by filtration, washed with methanol and dried in air. Yield: 63% (based on the Schiff base ligand). Analysis calculated for  $C_{12}H_{17}CuN_5O_2$ : C, 44.1; H, 5.2; N, 21.4%; found: C, 43.8; H, 5.4; N, 21.6%.

#### 2.4. Synthesis of [Co(HEP)(AE)]·Cl·H<sub>2</sub>O (2)

A methanol solution (10 mL) of copper(II) chloride (0.1 mmol, 17.0 mg) was added to a methanol solution (10 mL) of the Schiff base (0.1 mmol, 22.2 mg), 2-(2aminoethylamino)ethanol (0.1 mmol, 10.4 mg), and ammonium thiocyanate (0.1 mmol, 7.6 mg). The mixture was stirred at ambient conditions for 30 min to give a deep brown solution, which was allowed to evaporate slowly in air for 13 days, yielding brown single crystals, which were collected by filtration, washed with methanol, and dried in air. Yield: 32% (based on the Schiff base ligand). Analysis calculated for  $C_{17}H_{31}ClCoN_5O_4S$ : C, 41.2; H, 6.3; N, 14.1%; found: C, 41.5; H, 6.4; N, 13.8%.

#### 2.5. Crystal Structure Determination

The suitable single crystals of the complexes were mounted on glass fibers for data collection performed on a Bruker SMART 1000 CCD area diffractometer equipped with a graphite-monochromatic Mo K $\alpha$  radiation ( $\lambda = 0.71073$  Å) at 298 K. The unit cell dimensions were obtained with the least-squares refinements and the structures were solved by direct methods using SHELXTL-97 program.<sup>[11]</sup> The final refinement was performed by full-matrix least-squares methods with anisotropic thermal parameters for the non-hydrogen atoms on  $F^2$ . The amino H atoms in both complexes, and hydroxyl H atom of (2), were located from difference Fourier maps and refined isotropically, with N-H, O-H, and H...H distances restrained to 0.90(1), 0.85(1), and 1.43(2) Å, respectively. Other H atoms were placed in calculated positions and constrained to ride on their parent atoms. Multi-scan absorption correction was applied by using the SADABS program.<sup>[12]</sup> The crystallographic data and experimental details for structural analysis of the complexes are summarized in Table 1. Selected bond lengths and angles are given in Table 2. Hydrogen bonds are listed in Table 3. Crystallographic data for the complex has been deposited with the Cambridge Crystallographic Data Centre (CCDC 780758 (1) and 780759 for (2)).

#### 3. RESULTS AND DISCUSSION

The Schiff base ligand was first synthesized by Cros and coworkers using a Dean and Stark apparatus in benzene.<sup>[10]</sup> In

 TABLE 1

 Crystal data and refinement parameters for the complexes

Complex	(1)	(2)	
Formula	C <sub>12</sub> H <sub>17</sub> CuN <sub>5</sub> O <sub>2</sub> C <sub>17</sub> H <sub>31</sub> ClCoN <sub>5</sub> O		
FW	326.8	495.9	
Crystal shape/color	Block/blue	block/brown	
Crystal size/mm <sup>3</sup>	$0.32 \times 0.30 \times 0.27$	$0.32 \times 0.30 \times 0.27$	
Crystal system	Monoclinic	Triclinic	
Space group	$P2_1/c$ P-1		
a/Å	9.969(3)	7.205(2)	
b/Å	19.218(3)	9.976(3)	
c/Å	7.272(2)	16.737(5)	
$\alpha I^{\circ}$		98.522(4)	
$\beta$ / $^{\circ}$	108.788(3)	102.076(4)	
$\gamma I^{\circ}$		96.218(4)	
V/Å <sup>3</sup>	1319.0(6)	1151.4(6)	
Ζ	4	2	
$\lambda (MoK\alpha)/Å$	0.71073	0.71073	
T/K	298(2)	298(2)	
$\mu (MoK\alpha)/cm^{-1}$	1.665	0.984	
$T_{\min}$	0.6178	0.7437	
$T_{\rm max}$	0.6620	0.7771	
Measured reflections	10459	9350	
Unique reflections	2866	4869	
Observed reflections	2129	3121	
Parameters/restraints	186/1	279/7	
Goodness of fit on $F^2$	1.061	1.017	
$R_1, wR_2 [I \ge 2\sigma(I)]^a$	0.0424, 0.1116	0.0614, 0.1307	
$R_1, wR_2$ (all data) <sup>a</sup>	0.0636, 0.1286	0.1076, 0.1522	
$\frac{{}^{a}R_{1}}{\sum w(Fo^{2})^{2}]^{1/2}} = \sum_{i=1}^{n}   Fo   - \frac{1}{2}$	$ Fc  /\sum  Fo , wR_2$	$= \left[\sum w(Fo^2 - Fc^2)^2\right]$	

this paper, the Schiff base ligand was readily synthesized by condensation reaction of 1-(2-hydroxyphenyl)ethanone and 2-(2-aminoethylamino)ethanol in methanol with a beaker, with a very high yield.

The complexes were easily prepared by reaction of equimolar quantities of the metal salts, Schiff base ligand, 2-(2aminoethylamino)ethanol, and sodium azide or ammonium thiocyanate in methanol. We have intended to use 2-(2aminoethylamino)ethanol, sodium azide, and ammonium thiocyanate as bridging ligands when coordination, however, they acted as terminal ligands in both complexes. The 2-(2aminoethylamino)ethanol ligand only coordinates to the Co atom in a bidentate fashion. The terminal hydroxyl group of the Schiff base ligand coordinates to two Cu atoms in the dinuclear copper(II) complex reported by Cros and coworkers; however, in this paper, the hydroxyl groups in both complexes are not participate in coordination. This might be caused by the addition of the deprotonating agent triethylamine in the synthesis of the dinuclear copper(II) complex, and no such agents were used in the present work.

complexes (1) Cu1-01 1.857(2)Cu1-N1 1.965(3)Cu1-N2 1.989(3) Cu1-N3 1.983(3) 01-Cu1-N3 01-Cu1-N1 91.88(10) 89.96(12)N1-Cu1-N3 01-Cu1-N2 167.51(15) 175.89(11) N1-Cu1-N2 85.85(11) N3-Cu1-N2 92.99(12) (2) Co1-O1 1.880(3)Co1-N1 1.902(3)Co1-N2 1.996(4)Co1-N3 2.014(4)Co1-N4 1.960(4)Co1-N5 1.909(4)01-Co1-N1 93.83(13) 01-Co1-N5 91.69(15) N1-Co1-N5 90.43(15) 01-Co1-N4 84.99(15) N1-Co1-N4 176.65(17)N5-Co1-N4 92.73(16) 87.20(15) 01-Co1-N2 178.90(14)N1-Co1-N2 N5-Co1-N2 N4-Co1-N2 93.97(16) 88.71(16) O1-Co1-N3 87.88(15) N1-Co1-N3 91.39(15) N5-Co1-N3 178.15(15) N4-Co1-N3 85.44(16) N2-Co1-N3 91.69(16)

 TABLE 2

 Selected bond lengths (Å) and bond angles (°) for the

#### 3.1. IR Spectra

The Schiff base ligand and the two complexes show weak and sharp absorption bands in the region 3200–3300 cm<sup>-1</sup> corresponding to the  $\nu$ (–NH) of the amino groups. The weak and broad absorption bands in the region 3300–3600 cm<sup>-1</sup> are assigned to the phenolic groups of the Schiff base ligands and the water molecules. The strong and sharp band at 1643 for the Schiff base ligand is assigned to the  $\nu$ (C=N), which is shifted

 TABLE 3

 Hydrogen-bond geometry (Å,  $^{\circ}$ ) for the complexes

D–H···A	D–H	H···A	D···A	D–H· · · A
(1)				
$N2-H2A\cdots O1^i$	0.898(10)	2.32(3)	3.057(4)	139(4)
$O2H2 \cdots N5^i$	0.82	2.09	2.905(4)	171
(2)				
$O3\text{-}H3A{\cdots}O4^{ii}$	0.845(10)	1.969(14)	2.811(7)	173(6)
$N4H4A\cdots\text{-}O3^{iii}$	0.900(10)	2.36(4)	3.072(6)	136(4)
$N4H4B\cdots\text{-}C11^{iv}$	0.900(10)	2.48(3)	3.279(4)	148(4)
$N3-H3\cdots Cl1^v$	0.84(6)	2.72(6)	3.546(4)	166(5)
$N2-H2\cdot\cdot\cdot Cl1^v$	0.88(6)	2.35(6)	3.205(4)	162(5)
$O2H2A\cdots\text{-}Cl1^v$	0.82	2.35	3.111(5)	155

Symmetry codes: (i) x, 1/2 - y, 1/2 + z; (ii) 1 + x, y, 1 + z; (iii) -1 + x, y, z; (iv) x, 1 + y, z; (v) 1 + x, 1 + y, z.

to lower frequencies in the spectra of the complexes,  $1626 \text{ cm}^{-1}$  for (1) and  $1627 \text{ cm}^{-1}$  for (2). For (1), the strong absorption band indicative of the azide group is at 2027 cm<sup>-1</sup>. For (2), the strong absorption band indicative of the thiocyanate group is at 2093 cm<sup>-1</sup>. The spectra of the complexes also show some new bands in the region 500–400 cm<sup>-1</sup>, which reflect the formation of M–O and M–N bonds.<sup>[13–14]</sup>

#### **3.2.** Structure Description of (1)

The molecular structure of the complex (1) is shown in Figure 1. The Cu atom in the complex is four-coordinated by the phenolate O, imine N, and amine N atoms of the Schiff base ligand, and by one N atom of the azide ligand, forming a square planar geometry. All the bond lengths and angles are comparable to those observed in other copper(II) complexes with square



FIG. 1. The molecule of (1), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Mean diameter of zone of inhibition (mm) for the antimicrobial activity of the Schiff base and the complexes							
Compound	Pseudomonas aeruginosa	Staphylococcus aureus	Bacillus subtilis	Klebsiella pneumonia	Escherichia coli		
The Schiff base ligand	$8.3 \pm 0.6$	$12.5 \pm 0.8$	$7.0 \pm 0.5$	-	$5.4 \pm 0.5$		
(1)	$11.0 \pm 0.8$	$14.5\pm0.8$	$10.3\pm0.5$	$4.2 \pm 0.5$	$13.1 \pm 0.8$		
(2)	$13.3 \pm 0.6$	$21.7 \pm 1.0$	$16.3 \pm 0.8$	$8.3 \pm 0.8$	$23.2\pm1.0$		
Streptomycin	$12.1 \pm 0.8$	$25.0 \pm 1.0$	$23.3 \pm 1.0$	$15.5 \pm 1.0$	$17.2 \pm 1.0$		

 TABLE 4

 Mean diameter of zone of inhibition (mm) for the antimicrobial activity of the Schiff base and the complexes

planar coordination.<sup>[15–17]</sup> The coordination around the Cu atom displays somewhat distortion. The Cu atom deviates from the least-squares plane defined by the four basal donor atoms by 0.080(2) Å. The N1-Cu1-N2 bond angle is deviate from the value for an ideal square planar geometry by  $4.2(2)^{\circ}$ , which is caused by the strain created by the five-membered chelate ring Cu1-N1-C9-C10-N2.

In the crystal structure, molecules are linked through intermolecular N-H···O and O-H···N hydrogen bonds, to form chains running along the *c* axis, as shown by Figure 2.

#### 3.3. Structure Description of (2)

The molecular structure of the complex (2) is shown in Figure 3. The complex consists of a  $[Co(HEP)(AE)(NCS)]^+$  cation, a chloride anion, and a water molecule of crystallization. The Co atom in the cation is six-coordinate in an octahedral geometry, with one phenolate O, one imine N and

one amine N atoms of the Schiff base ligand, and one primary amine N atom of 2-(2-aminoethylamino)ethanol defining the equatorial plane, and with one secondary amine N atom of 2-(2-aminoethylamino)ethanol and one thiocyanate N atom occupying the two axial positions. All the bond lengths related to the Co atom are comparable with those observed in other similar cobalt(III) complexes with Schiff bases.<sup>[18–20]</sup> The octahedral coordination around the Co atom displays somewhat distortion. The Co atom deviates from the least-squares plane defined by the four equatorial donor atoms by 0.030(2) Å toward N5. The N1-Co1-N2 and N3-Co1-N4 bond angles are deviate from the values for an ideal octahedral geometry by 2.8(2) and 4.6(2)°, respectively, which are caused by the strain created by the five-membered chelate rings Co1-N1-C9-C10-N2 and Co1-N3-C14-C13-N4.

In the crystal structure, molecules are linked through intermolecular  $O-H\cdots O$ ,  $N-H\cdots O$ ,  $N-H\cdots Cl$  and  $O-H\cdots Cl$ 



FIG. 2. The molecular packing of (1). Intermolecular hydrogen bonds are shown as dashed lines.



FIG. 3. The molecule of (2), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

hydrogen bonds, to form chains running along the a axis, as shown by Figure 4.

#### 3.4. Antibacterial Activities

Antibacterial activities of the complexes were tested by the well diffusion method using Sabouraud dextrose agar and Müller Hinton agar.<sup>[21]</sup> The zone of inhibition was recorded on the completion of the incubation and the mean diameter for each complex at 100  $\mu$ g mL<sup>-1</sup> was recorded. The zone of inhibition was recorded in diameters. Stock solutions of tested compounds were prepared in dimethyl sulfoxide. The diameters of the zone of inhibition produced by the compounds were compared with the standard antibiotics Streptomycin 10  $\mu$ g per well. Each test was carried out for three times to minimize the error.

The Schiff base and the two complexes were subjected to antibacterial activity against Pseudomonas aeruginosa, Staphylococcus aureus, Bacillus subtilis, Klebsiella pneumonia, and Escherichia coli. The screening results are summarized in Table 4. It can be seen that both complexes have stronger activities than those of the Schiff base ligand, and the cobalt(III) complex (2)has stronger activities than those of the copper(II) complex (1). Such an increased activity of the complexes can be explained on the basis of Overtone's concept<sup>[22]</sup> and Tweedy's Chelation theory<sup>[23]</sup> that the metal complexes have a better activity than the free ligands. On chelating, the polarity of the metal ion will be reduced to a greater extent due to the overlap of the ligand orbital and partial sharing of positive charge of the metal ion with donor groups. Further, it increases the delocalization of  $\pi$ -electrons over the whole chelate ring and enhances the lipophilicity of the complex. This increased lipophilicity enhances the



FIG. 4. The molecular packing of (2), viewed along the b axis. Intermolecular hydrogen bonds are shown as dashed lines.

penetration of the complexes into lipid membrane and blocks the metal binding sites on enzymes of micro-organisms.

#### 4. CONCLUSIONS

In this article, two new copper(II) and cobalt(III) complexes with Schiff base ligands were synthesized and characterized by elemental analysis, IR spectra, and single crystal X-ray diffraction. The in vitro antimicrobial test shows that both complexes have stronger activities than those of the Schiff bases.

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