ISSN 1070-3284, Russian Journal of Coordination Chemistry, 2017, Vol. 43, No. 3, pp. 181–188. © Pleiades Publishing, Ltd., 2017.

Synthesis, Crystal Structures, and Antibacterial Activity of Copper(II) and Cobalt(III) Complexes Derived from 2-[(2-Dimethylaminoethylimino)methyl]-4-Methylphenol[1]

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Received January 7, 2016

Abstract—A new tetranuclear copper(II) complex (**I**) and a new mononuclear cobalt(III) complex (**II**) have been synthesized from the Schiff base compound 2-[(2-dimethylaminoethylimino)methyl]-4-methylphenol. The complexes have been characterized by physico-chemical and spectroscopic methods, as well as single crystal X-ray determination (CIF files CCDC nos. 1447778 (**I**) and 1447779 (**II**)). The Cu atoms in complex **I** are in square pyramidal coordination, and the Co atom in complex **II** is in octahedral coordination. Crystal structures of the complex are stabilized by hydrogen bonds and $\pi\cdots\pi$ interactions. The complexes and the Schiff base compound were assayed for antibacterial activities against three Gram-positive bacterial strains (*B. subtilis*, *S. aureus*, and *St. faecalis*) and three Gram-negative bacterial strains (*E. coli*, *P. aeruginosa*, and *E. cloacae*) by MTT method. As a result, the complexes showed effective antimicrobial activity against the microorganisms tested.

Keywords: Schiff base, copper(II) complex, cobalt(III) complex, X-ray diffraction, hydrogen bonds, antibacterial activity

DOI: 10.1134/S107032841703006X

INTRODUCTION

Schiff bases represent one of the most widely utilized classes of ligands in metal coordination chemistry. They offer versatile and flexible ligands capable of binding various metal ions to give complexes with versatile structures and properties [1–5]. Over the past few decades considerable study has been made on the chemistry of copper(II) and cobalt(III) complexes derived from Schiff base ligands due to their important role in catalytic, magnetic and biological properties [6–13]. An important aspect of Cu(II) and Co(III) salen type complexes is their antibacterial application [14–18]. We report here the synthesis, characterization including single crystal X-ray structure of a new copper(II) complex $[Cu_4(L)_2(N_3)_4Cl_2(H_2O)_2]$ (**I**) and a new cobalt(III) complex $[Co(L)(HL)(N_3)]ClO_4$ (**II**), where HL is 2-[(2-dimethylaminoethylimino)methyl]-4-methylphenol. The antibacterial activity against three Gram-positive bacterial strains (*B. subtilis*, *S. aureus*, and *St. faecalis*) and three Gram-negative bacterial strains (*E. coli*, *P. aeruginosa*, and *E. cloacae*) by MTT method was studied.

EXPERIMENTAL

Materials and physical methods. The Schiff base compound HL was prepared by 1 : 1 condensation of 5-methylsalicylaldehyde and *N*,*N*-dimethylethane-1,2-diamine in methanol, according to the literature method [19]. All the other reagents and solvents were purchased from commercial sources and used as received. FT-IR spectra were recorded as KBr pellets on Bruker Tensor-27. Elemental (C, H, and N) analyses were performed on a Perkin-Elmer 2400 II analyzer. Single crystal X-ray diffraction was carried out with a Bruker Apex II CCD diffratometer. Molar conductivity of the complexes in acetonitrile was measured with a DDS-11A molar conductivity meter.

Caution! Perchlorate and azide complexes of metal ions are potentially explosive. Only a small amount of material should be prepared, and it should be handled with caution.

Synthesis of complex I. A methanol solution (20 mL) of copper(II) chloride dihydrate (0.170 g, 1.00 mmol) was added dropwise to a stirred solution of HL (0.206 g, 1.00 mmol) and sodium azide (0.130 g, 2.00 mmol) in methanol (20 mL). After a few minutes a blue precipitate started to form. It was dissolved by adding the requisite amount of acetonitrile. After one-hour stirring, the solution was filtered and the filtrate

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was allowed to slowly evaporate. Deep blue single crystals suitable for X-ray diffraction studies that precipitated within a few days were collected by filtration and washed with methanol. The yield was 212 mg (45%).

For $C_{24}H_{38}N_{16}O_4Cl_2Cu_4$

anal. calcd., %:	C, 30.67;	H, 4.08;	N, 23.85.
Found, %:	C, 30.51;	H, 4.15;	N, 23.96.

Synthesis of complex II. A methanol solution (20 mL) of cobalt(III) perchlorate hexahydrate (0.366 g, 1.00 mmol) was added dropwise to a stirred solution of HL (0.206 g, 1.00 mmol) and sodium azide (0.130 g, 2.00 mmol) in methanol (20 mL). After a few minutes a brown precipitate started to form. It was dissolved by adding the requisite amount of acetonitrile. After one-hour stirring, the solution was filtered and the filtrate was allowed to slowly evaporate. Brown single crystals suitable for X-ray diffraction studies that precipitated within a few days were collected by filtration and washed with methanol. The yield was 172 mg (56%).

For $C_{24}H_{35}N_7O_6ClCo$

anal. calcd., %:	C, 47.10;	H, 5.76;	N, 16.02.
Found, %:	C, 47.27;	H, 5.85;	N, 15.91.

X-ray structure determination. Intensity data of the complexes were collected at 298(2) K on a Bruker Apex II CCD diffractometer using graphite-monochromated MoK_α radiation ($\lambda = 0.71073$ Å). For data processing and absorption correction the packages SAINT and SADABS [20] were used. The structures were solved by direct and Fourier methods and refined by full-matrix least-squares based on F^2 using SHELXL-97 [21] package. The non-hydrogen atoms were refined anisotropically. The hydrogen atoms have been placed at geometrical positions with fixed thermal parameters. The perchlorate anion of complex **II** is disordered over two sites and modeled accordingly, with occupancies of 0.613(3) and 0.387(3). Crystallographic data of the complexes are summarized in Table 1. Selected bond lengths and angles are listed in Table 2.

Supplementary material for structures has been deposited with the Cambridge Crystallographic Data Centre (CCDC nos. 1447778 for **I** and 1447779 for **II**; deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/conts/retrieving.html).

Antibacterial activity. Antibacterial activity of the complex was tested against *B. subtilis*, *S. aureus*, *S. faecalis*, *P. aeruginosa*, *E. coli*, and *E. cloacae* using MTT (3-(4,5-dimethyl-2-thiazolyl)-2,5-diphenyl-2-*H*-tetrazolium bromide) medium. The minimum inhibitory concentrations (MICs) of the complexes were determined by a colorimetric method using MTT dye [22]. A stock solution of the complexes (50 μg mL^{-1}) in DMSO was prepared and quantities of the complexes were incorporated in specified quantity of sterilized liquid medium. A specified quantity of the medium containing the complexes was poured into microtitration plates. Suspension of the microorganism was prepared to contain approximately 10^5 cfu mL^{-1} and applied to microtitration plates with serially diluted complexes in DMSO to be tested, and incubated at 37°C for 24 h for bacteria. After the MICs were visually determined on each microtitration plates, 50 μL of phosphate buffered saline (PBS 0.01 mol L^{-1}, pH 7.4: $Na_2HPO_4 \cdot 12H_2O$ 2.9 g, KH_2PO_4 0.2 g, NaCl 8.0 g, KCl 0.2 g, distilled water 1000 mL) containing 2 mg mL^{-1} of MTT was added to each well. Incubation was continued at room temperature for 4–5 h. The content of each well was removed, and 100 μL of isopropanol containing 5% 1 mol L^{-1} HCl was added to extract the dye. After 12 h of incubation at room temperature, the optical density was measured with a microplate reader at 570 nm.

RESULTS AND DISCUSSION

Reaction of HL with copper(II) chloride and cobalt(II) perchlorate, respectively, in the presence of sodium azide produces the tetranuclear copper(II) complex and mononuclear cobalt(III) complex. Clearly, aerial oxidation of cobatl(II) to cobalt(III) and metal assisted deprotonation of the phenolic moieties took place during the formation of the complexes. The poor conductivity of complex **I** (21 Ω^{-1} cm^2 mol^{-1}) indicates the non-electrolytic nature. The conductivity of complex **II** is 125 Ω^{-1} cm^2 mol^{-1}, suggests the 1 : 1 electrolytic nature. The characteristic imine stretching of the complexes is observed at 1631 cm^{-1} for **I** and 1628 cm^{-1} for **II** as strong signal. Appearance of intense bands at 2066 cm^{-1} for **I** and 2045 cm^{-1} for **II** indicates the presence of azide ligands. The IR spectrum of complex **I** exhibits one broad and weak absorption centered at 3450 cm^{-1} due to the vibration of the hydroxyl groups of the water ligands. The IR spectrum of complex **II** exhibits one sharp and weak absorption at 3217 cm^{-1} due to the vibration of the NH group of the Schiff base ligand.

The crystal structure of complex **I** is shown in Fig. 1a. The complex possesses a crystallagraphic inversion center symmetry. The outer Cu atoms are five-coordinated, with one oxygen and two nitrogen atoms of the Schiff base ligand, and one azido nitrogen atom in the equatoiral plane, and with one water oxygen atom defining the apical position. The inner Cu atoms are also five-coordinated, with one oxygen atom of the Schiff base ligand, one Cl atom, and two azido nitrogen atoms in the equatoiral plane, and with one azido nitrogen atom defining the apical position. The coordination environment of the metal ions is a

Table 1. Crystallographic data and refinement parameters for complexes **I** and **II**

Parameter	Value	
	I	**II**
Molecular weight	939.76	611.97
Crystal color, habit	Blue, block	Brown, block
Crystal size, mm	0.27 × 0.25 × 0.25	0.28 × 0.26 × 0.26
Crystal system	Monoclinic	Orthorhombic
Space group	$P2_1/n$	$P2_12_12_1$
Unit cell dimensions:		
a, Å	12.4205(12)	7.8385(3)
b, Å	12.5819(9)	14.9999(7)
c, Å	12.5614(15)	24.1656(11)
β, deg	97.966(2)	
V, Å^3	1944.1(3)	2841.3(2)
Z	2	4
ρ_{calcd}, g cm^{-3}	1.605	1.431
μ, mm^{-1}	2.348	0.749
θ Range collected, deg	2.97–25.49	2.87–25.50
$T_{\min}$ and $T_{\max}$	0.5698 and 0.5914	0.8178 and 0.8291
Reflections collected	8257	12897
Unique reflections	3446	5221
Observed reflections ($I \geq 2\sigma(I)$)	1893	4596
Restraints/parameters	24/235	43/399
R_1, wR_2 ($I \geq 2\sigma(I)$)	0.0812, 0.1844	0.0459, 0.1136
R_1, wR_2 (all data)	0.1437, 0.2434	0.0549, 0.1208
GOOF	1.033	1.036
Largest differences in peak/hole, e/Å^3	1.154/–0.806	0.266/–0.628

slightly distorted square pyramidal. The average deviations of the equatorial donor atoms for the outer and inner Cu coordination are 0.0962 and 0.2225 Å, and the displacements of the Cu(1) and Cu(2) centers from the corresponding least-squares planes are 0.212 and 0.149 Å, respectively. The transoid angles in the equatorial planes (159°–173°) and the cisoid angles (79°–99°) deviate somewhat from the ideal values. The coordinate bond values are comparable to those in copper(II) complexes with Schiff bases [23–25].

As shown in Fig. 2a, the complex molecules are linked through hydrogen bonds (Table 3), as well as π···π interactions (Cg(1)···Cg(2)^i 4.962(3), Cg(2)···Cg(2)^i 4.739(3) Å, ^{i} $1 - x$, $-y$, $1 - z$; Cg(1) and Cg(2) are the centroids of Cu(1)–O(1)–C(2)–C(1)–C(8)–N(1) and C(1)–C(2)–C(3)–C(4)–C(5)–C(6), respectively).

The component and crystal structure of complex **II** are shown in Fig. 1b. The asymmetric unit of the complex contains a mononuclear $[Co(L)(HL)(N_3)]^+$ cation and a disordered perchlorate anion. The Co atom is six-coordinated, with one oxygen and two nitrogen atoms of a deprotonated Schiff base ligand L, and one imino nitrogen atom of a zwitterionic Schiff base ligand HL in the equatoiral plane, and with one phenolate oxygen atom of HL and one azido nitrogen atom defining the axial positions. The coordination environment of the metal ion is slightly distorted octahedral. The average deviation (0.0291 Å) of the equatorial donor atoms and the displacement (0.0104 Å) of the metal center from the least-squares plane O(1)–O(3)–O(4)–N(1) indicates that the N_2O_2 cavity affords an almost perfect plane to the metal center. The transoid angles (173.03(14)°, 177.91(15)°, and 177.67(15)°) and the cisoid angles (84.78(13)°–95.03(13)°) deviate slightly from the ideal values. The

Table 2. Selected bond distances (Å) and angles (deg) for complexes **I** and **II**

Bond	*d*, Å	Bond	*d*, Å
		I	
Cu(1)–O(1)	1.942(7)	Cu(1)–N(1)	1.942(8)
Cu(1)–N(2)	2.049(8)	Cu(1)–N(6)	2.158(6)
Cu(1)–O(2)	2.369(8)	Cu(2)–N(3)	1.966(8)
Cu(2)–O(1)	1.967(6)	Cu(2)–N(3A)	2.030(8)
Cu(2)–Cl(1)	2.251(3)		
		II	
Co(1)–O(1)	1.891(3)	Co(1)–O(2)	1.919(3)
Co(1)–N(1)	1.956(3)	Co(1)–N(3)	1.899(3)
Co(1)–N(4)	2.040(4)	Co(1)–N(5)	1.950(4)
Angle	ω, deg	Angle	ω, deg
		I	
O(1)Cu(1)N(1)	90.0(3)	O(1)Cu(1)N(2)	172.7(3)
N(1)Cu(1)N(2)	85.9(3)	O(1)Cu(1)N(6)	89.0(2)
N(1)Cu(1)N(6)	162.6(3)	N(2)Cu(1)N(6)	93.0(3)
O(1)Cu(1)O(2)	89.8(3)	N(1)Cu(1)O(2)	98.5(3)
N(2)Cu(1)O(2)	96.8(3)	N(6)Cu(1)O(2)	98.9(3)
N(3)Cu(2)O(1)	170.0(3)	N(3)Cu(2)N(3A)	79.9(3)
O(1)Cu(2)N(3A)	92.1(3)	N(3)Cu(2)Cl(1)	95.9(2)
O(1)Cu(2)Cl(1)	93.6(2)	N(3)Cu(2)Cl(1A)	159.2(3)
		II	
O(1)Co(1)N(3)	84.78(13)	O(1)Co(1)O(2)	88.24(12)
N(3)Co(1)O(2)	95.03(13)	O(1)Co(1)N(5)	173.03(14)
N(3)Co(1)N(5)	88.76(15)	O(2)Co(1)N(5)	89.72(15)
O(1)Co(1)N(1)	94.21(13)	N(3)Co(1)N(1)	177.91(15)
O(2)Co(1)N(1)	86.76(13)	N(5)Co(1)N(1)	92.33(15)
O(1)Co(1)N(4)	94.08(14)	N(3)Co(1)N(4)	85.43(16)
O(2)Co(1)N(4)	177.67(15)	N(5)Co(1)N(4)	88.00(16)
N(1)Co(1)N(4)	92.82(15)		

coordinate bond values are comparable to those in cobalt(III) complexes with Schiff bases [26–28].

As shown in Fig. 2b, the complex molecules and the perchlorate anions are linked through hydrogen bonds (Table 3), as well as π···π interactions (Cg(3)···Cg(4) 4.976(3), Cg(4)···Cg(5)^{ii} 4.804(3) Å, ^{ii} $-x$, $-1/2 + y$, $1/2 - z$; Cg(3), Cg(4) and Cg(5) are the centroids of Co(1)–O(1)–C(2)–C(1)–C(8)–N(1), C(13)–C(14)–C(15)–C(16)–C(17)–C(18) and C(1)–C(2)–C(3)–C(4)–C(5)–C(6), respectively).

The complexes and HL were screened for antibacterial activities against three Gram-positive bacterial strains (*B. subtilis*, *S. aureus*, and *St. faecalis*) and three Gram-negative bacterial strains (*E. coli*, *P. aeruginosa*, and *E. cloacae*) by MTT method. The MICs of the complexes against the bacteria are presented in Table 4. Penicillin G was tested as a reference drug. The antibacterial activities of the complexes are obvious higher than HL. The free Schiff base is inactive against both the Gram-positive and Gram-negative bacterial strains except for *S. aureus* whish shows weak activity. Complex **I** exhibited significant activities against *B. subtilis*, *S. aureus* and *E. cloacae*, and medium activity against *St. faecalis*, *P. aeruginosa* and *E. coli*. Complex **II** exhibited significant activities

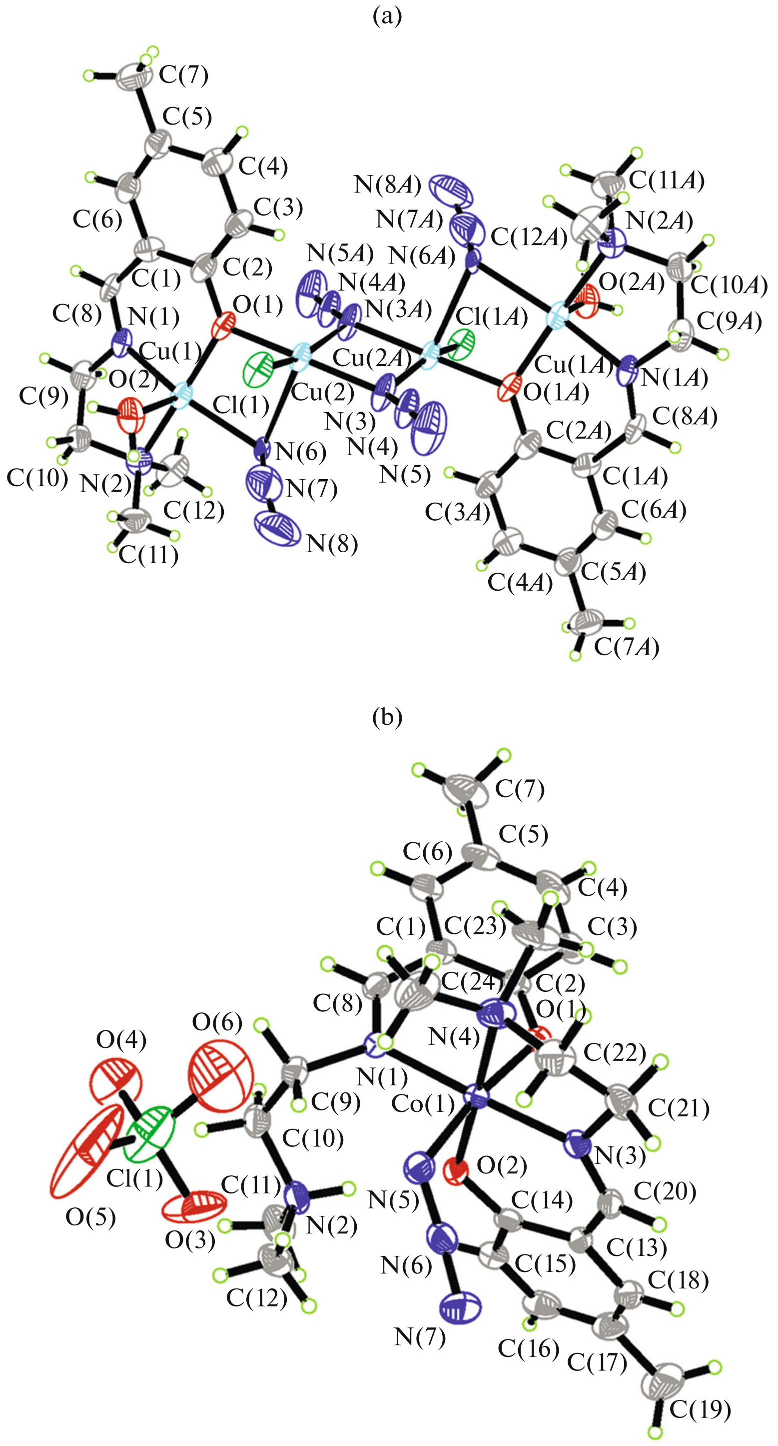

Fig. 1. Molecular structure of complex **I** (a) and complex **II** (b) with 30% thermal ellipsoids. Atoms labeled with the suffix *A* in **I** are related to the symmetry operation $1 - x$, $1 - y$, $1 - z$.

against *B. subtilis* and *S. aureus*, and medium activity against *St. faecalis* and *E. cloacae*, while weak or no activity against the Gram negative bacterial strains. Such an enhanced activity of the complexes than the free Schiff base can be explained on the basis of Overtone's concept and Tweedy's chelation theory [26]. According to Overtone's concept of cell permeability, the lipid membrane that surrounds the cell favors the passage of only lipid soluble materials due to which liposolubility is an important factor that controls antimicrobial activity. On chelation, the polarity of the metal ion is reduced to a greater extent due to the over-

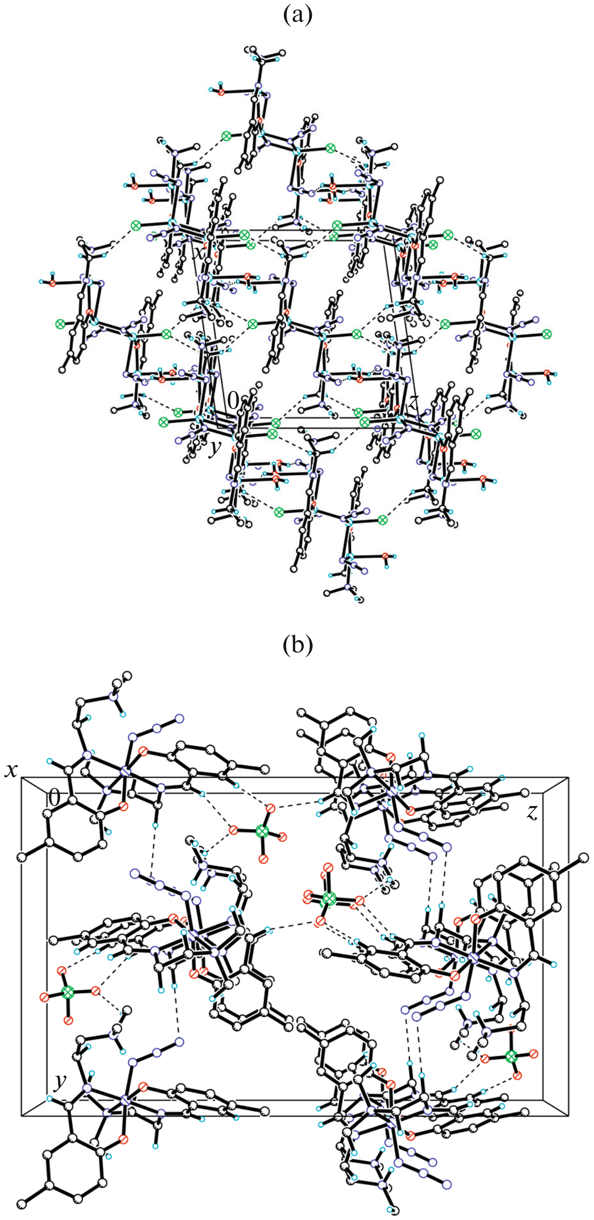

Fig. 2. Molecular packing diagram of complex **I** (a), viewed along the *y* axis direction and complex **II** (b), viewed along the *x* axis direction. Hydrogen bonds are drawn as dotted lines.

lap of the ligand orbital and partial sharing of the positive charge of the metal ion with donor groups. Further, it increases the delocalisation of π-electrons over the whole chelate ring and enhances the lipophilicity of the complexes. This increased lipophilicity enhances the penetration of the complexes into lipid membranes and blocking of metal binding sites on the enzymes of the microorganisms. The complexes also disturb the respiration process of the cell and thus block the synthesis of the proteins that restricts further growth of the organism. The variation in the effectiveness of the different compounds against different organisms depends on the impermeability of the cells of microbes or difference in ribosome of the microbial cells.

Thus, a new tetranuclear copper(II) complex and a new mononuclear cobalt(III) complex have been synthesized from the Schiff base compound 2-[(2-dimethylaminoethylimino)methyl]-4-methylphenol and characterized. Crystal structures of the complexes are stabilized by hydrogen bonds and π···π interactions. The antibacterial assay of the free Schiff base and the

Table 3. Distances (Å) and angles (deg) involving hydrogen bonding of complexes **I** and **II***

Contact D–H···A	Distance, Å			Angle D–H···A, deg
	D–H	H···A	D···A	
		I		
O(2)–H(2B)···N(8)$^{\#1}$	0.85	2.27(6)	3.02(2)	146(9)
C(9)–H(9A)···Cl(1)$^{\#1}$	0.97	2.81(6)	3.558(7)	135(8)
C(9)–H(9B)···Cl(1)$^{\#2}$	0.97	2.78(5)	3.638(8)	148(8)
C(11)–H(11C)···N(7)	0.96	2.58(5)	3.259(8)	128(7)
		II		
N(2)–H(2)···O(2)	0.90	1.86(1)	2.753(4)	172(5)
N(2)–H(2)···N(1)	0.90	2.61(5)	3.071(5)	112(4)
C(8)–H(8)···O(5)$^{\#3}$	0.93	2.45(2)	3.229(4)	141(3)
C(9)–H(9B)···N(5)	0.97	2.29(3)	2.929(4)	123(3)
C(12)–H(12A)···O(3)$^{\#4}$	0.96	2.42(3)	3.194(5)	138(4)
C(18)–H(18)···O(5)	0.93	2.30(3)	3.225(5)	178(5)
C(20)–H(20)···O(3)	0.93	2.42(3)	3.299(4)	158(4)
C(21)–H(21A)···N(7)$^{\#5}$	0.97	2.61(3)	3.573(5)	172(5)
C(22)–H(22B)···N(5)	0.97	2.61(3)	3.015(4)	105(4)
C(23)–H(23C)···O(1)	0.96	2.46(3)	2.843(3)	104(4)
C(24)–H(24A)···N(5)	0.96	2.37(3)	2.979(4)	121(5)

* Symmetry codes: $^{\#1}$ $1 - x, -y, -z$; $^{\#2}$ $1 - x, 2 - y, -z$; $^{\#3}$ $1/2 - x, 1 - y, 1/2 + z$; $^{\#4}$ $1 - x, -1/2 + y, 1/2 - z$; $^{\#5}$ $1 - x, 1/2 + y, 1/2 - z$.

Table 4. MICs (μg mL^{-1}) of complexes **I**, **II** and related material

Tested material	Gram positive			Gram negative		
	B. subtilis	*S. aureus*	*St. faecalis*	*P. aeruginosa*	*E. coli*	*E. cloacae*
I	3.12	0.78	12.5	12.5	12.5	6.25
II	6.25	3.12	12.5	>50	>50	25
HL	>50	25	>50	>50	>50	>50
Penicillin G	1.56	1.56	1.56	6.25	6.25	3.12

complexes shows that the complexes are more active compared to the Schiff base. The complexes have effective activity against some bacterial strains.

ACKNOWLEDGMENTS

Financial support from the Third Affiliated Hospital of Soochow University is gratefully acknowledged.

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