

Syntheses, Characterization, and Antibacterial Property of Polynuclear Cobalt(III) and Copper(II) Complexes Derived from Similar Tridentate Schiff Bases¹

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Abstract—An end-on azido-bridged trinuclear cobalt(III) complex $[\text{Co}_3(\text{L}^1)_2(\mu_{1,1}\text{-N}_3)_4(\text{N}_3)_2(\text{OMe})(\text{MeOH})]$ (**I**) and a phenolato-bridged dinuclear copper(II) complex $[\text{Cu}_2(\text{L}^2)_2(\text{NCS})_2]$ (**II**), where L^1 is the deprotonated form of 2-((2-(dimethylamino)ethylimino)methyl)-4-fluorophenol, and L^2 is the deprotonated form of 2-((3-(dimethylamino)propylimino)methyl)-4-fluorophenol, have been prepared and characterized by elemental analyses, IR and UV-Vis spectra, and single crystal X-ray diffraction (CIF files CCDC nos. 1023376 (**I**); 1023377 (**II**)). The Co atoms in complex **I** are in octahedral coordination, and the Cu atoms in complex **II** are in square pyramidal coordination. The antibacterial properties have been tested on some bacteria and yeast.

Keywords: Schiff base, cobalt complex, copper complex, crystal structure, antibacterial property

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INTRODUCTION

Schiff bases have been extensively used as ligands to construct complexes with various metal ions. To date, numerous Schiff bases and their complexes have been prepared and studied their properties, such as magnetic [1–5], catalytic [6–10], luminescence [11], as well as biological applications [12–15]. Considerable attention has been focused on the polynuclear transition metal complexes due to their wide applications in magnetic fields [16–19]. The preferred way to construct polynuclear complexes is the use of suitable bridging groups, such as N_3^- , NCS^- , $\text{N}(\text{CN})_2^-$, dicarboxylate, 4,4'-Bipy, etc. [20–25]. Among the bridging groups, azide anions have the most versatile coordination modes, including $\mu_{1,1}$ - (end-on, EO), $\mu_{1,3}$ - (end-to-end, EE), $\mu_{1,1,3}$ -, $\mu_{1,1,3,3}$ -, etc. As for the thiocyanate anions, the coordination modes are much simpler than azide anions, which usually adopt $\mu_{1,1}$ - and $\mu_{1,3}$ -bridging modes. Schiff bases are among the most popular ligands in coordination chemistry. Polynuclear complexes with Schiff bases exhibit interesting magnetic properties [17, 18, 26–30]. Tridentate Schiff bases with phenolate-O, imino-N, and amino-N donor atoms can block three coordination sites at the metal atoms, leaving the

others available for the interaction with various other ligands, such as azide or thiocyanate. In the present work, the syntheses and structures of an end-on azido-bridged trinuclear cobalt(III) complex $[\text{Co}_3(\text{L}^1)_2(\mu_{1,1}\text{-N}_3)_4(\text{N}_3)_2(\text{OMe})(\text{MeOH})]$ (**I**), and a phenolato-bridged dinuclear copper(II) complex $[\text{Cu}_2(\text{L}^2)_2(\text{NCS})_2]$ (**II**), where L^1 is the deprotonated form of 2-((2-(dimethylamino)ethylimino)methyl)-4-fluorophenol and L^2 is the deprotonated form of 2-((3-(dimethylamino)propylimino)methyl)-4-fluorophenol, are presented.

EXPERIMENTAL

Materials and methods. Starting materials, reagents and solvents were purchased from commercial suppliers and used as received. Elemental analyses were performed on a Perkin-Elmer 240C elemental analyzer. IR spectra were recorded on a Jasco FT/IR-4000 spectrometer as KBr pellets in the 4000–400 cm^{-1} region. UV-Vis spectrum was recorded on a Lambda 900 spectrometer. Thermal stability analysis was performed on a Perkin-Elmer Pyris Diamond TG-DTA thermal analyses system. Single crystal X-ray diffraction was carried out on a Bruker SMART 1000 CCD diffractometer.

Caution! Although our samples never exploded during handling, metal azide complexes are poten-

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tially explosive. Only a small amount of material should be prepared and it should be handled with care.

Synthesis of I. 5-Fluorosalicylaldehyde (1.40 g, 0.01 mol) and *N,N*-dimethylethane-1,2-diamine (0.88 g, 0.01 mol) were mixed in methanol (50 mL). The mixture was stirred at ambient temperature for 30 min to give a yellow solution. To the solution was added an aqueous solution (10 mL) of sodium azide (1.95 g, 0.03 mol) and a methanol solution (30 mL) of cobalt chloride hexahydrate (4.76 g, 0.02 mol). The mixture was further stirred for 30 min to give a brown solution. Single crystals of the complex, suitable for X-ray diffraction, were obtained after a week. The yield was 1.78 g (39%) on the basis of 5-fluorosalicylaldehyde.

IR data (ν , cm^{-1}): 3506 m (OH), 2057 s ($\mu_{1,1}$ -N₃), 2019 s (N₃), 1636 s (C=N), 1552 w, 1469 m, 1392 w, 1296 m, 1147 w, 1073 w, 965 w, 907 w, 871 w, 822 w, 778 w, 478 w, 434 w. UV (λ , nm (ϵ , $\text{L mol}^{-1} \text{cm}^{-1}$)): 250 (1.67×10^4), 339 (6.08×10^3), 407 (3.98×10^3), 500 (1.99×10^3).

For C₂₄H₃₅N₂₂O₄F₂Co₃

anal. calcd., %	C, 31.7	H, 3.9	N, 33.8
Found, %	C, 31.5	H, 4.0	N, 34.0

Synthesis of II. 5-Fluorosalicylaldehyde (1.40 g, 0.01 mol) and *N,N*-dimethylpropane-1,3-diamine (0.102 g, 0.01 mol) were mixed in methanol (50 mL). The mixture was stirred at ambient temperature for 30 min to give a yellow solution. To the solution was added an aqueous solution (10 mL) of ammonium thiocyanate (2.28 g, 0.03 mol) and a methanol solution (30 mL) of copper acetate monohydrate (3.98 g, 0.02 mol). The mixture was further stirred for 30 min to give a blue solution. Single crystals of the complex, suitable for X-ray diffraction, were obtained after a week. The yield was 1.53 g (44%) on the basis of 5-fluorosalicylaldehyde.

IR data (ν , cm^{-1}): 2098 s (NCS), 1632 s (C=N), 1553 m, 1465 s, 1386 w, 1287 m, 1245 w, 1213 w, 1145 w, 1056 w, 1010 w, 957 w, 872 w, 821 w, 795 m, 554 w, 518 w, 450 w. UV (λ , nm (ϵ , $\text{L mol}^{-1} \text{cm}^{-1}$)): 240 (2.16×10^4), 269 (1.54×10^4), 305 (6.30×10^3), 385 (7.28×10^3).

For C₂₆H₃₂N₆O₂S₂F₂Cu₂

anal. calcd., %	C, 45.3	H, 4.7	N, 12.2
Found, %	C, 45.1	H, 4.6	N, 12.3

X-ray crystallography. Single crystal X-ray data for the complexes were collected on a Bruker SMART APEX CCD diffractometer using the SMART/SAINT software [31]. Intensity data were collected using graphite-monochromatized MoK α radiation (0.71073 Å) at 298(2) K. The structures were

solved by direct methods using SHELX-97 [32]. Empirical absorption corrections were applied with SADABS [33]. All non-hydrogen atoms were refined with anisotropic displacement coefficients. The methanol H atom of complex I was located from a difference Fourier map and refined isotropically, with O–H distance restrained to 0.85 Å. The remaining hydrogen atoms bonded to carbon and nitrogen were included in geometric positions and given thermal parameters equivalent to 1.2 times those of the atom to which they were attached. Crystallographic data and refinement parameters are given in Table 1, and important interatomic distances and angles are given in Table 2.

Supplementary material for structures have been deposited with the Cambridge Crystallographic Data Centre (CCDC nos. 1023376 (I); 1023377 (II); deposit@ccdc.cam.ac.uk or <http://www.ccdc.cam.ac.uk>).

Biological assay. The antibacterial property of the complexes was evaluated by a macro-dilution method using *Staphylococcus aureus*, *Escherichia coli*, and the yeasts *Candida parapsilosis*. The cultures of bacteria and yeasts were incubated under vigorous shaking. The compounds were dissolved in small amount of DMSO. Concentration of the tested compounds ranging from 0.01 to 2.50 mmol L⁻¹ for the bacteria and yeasts was used in all experiments. The antibacterial activity was characterized by IC₅₀ and MIC values. MIC experiments on subculture dishes were used to assess the minimal microbicidal concentration (MMC). Subcultures were prepared separately in Petri dishes containing competent agar medium and incubated at 30 °C for 48 h. The MMC value was taken as the lowest concentration, which showed no visible growth of microbial colonies in the subculture dishes.

RESULTS AND DISCUSSION

The molecular structure of complex I is shown in Fig. 1a. The complex is an end-on azido-bridged trinuclear cobalt(III) species. The Co(1) atom is hexacoordinated with an octahedral geometry, the axial positions being occupied by one methanol O(3) atom and one azido N(8) atom. The equatorial plane is defined by three azido atoms N(5), N(11), N(14) and one deprotonated methanol O(4) atom. Both Co(2) and Co(3) atoms are also hexacoordinated with an octahedral geometry, the axial positions being occupied by one bridging azido N(5) atom for Co(2), N(11) for Co(3) and one terminal azido N(17) atom for Co(2), N(20) for Co(3). The equatorial planes are defined by three donor atoms of the Schiff base ligands and the other bridging azido N atoms. The distances of Co(1)⋯Co(2) and Co(1)⋯Co(3) are about 3.26 Å. The axial bond angles of the octahedral coordination are vary from 170.1(4)° to 172.1(5)°, indicating the octahedral coordination are distorted. This is mainly caused by the strain created by the four-membered

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

Parameter	Value	
	I	II
F_w	910.5	689.8
Crystal shape; colour	Block; brown	Block; blue
Crystal size, mm	$0.17 \times 0.13 \times 0.12$	$0.23 \times 0.22 \times 0.20$
Crystal system	Orthorhombic	Triclinic
Space group	$Pca2_1$	$P\bar{1}$
a , Å	15.285(2)	11.2952(7)
b , Å	8.330(1)	12.0782(7)
c , Å	28.737(2)	12.1290(7)
α , deg	90	75.379(2)
β , deg	90	66.515(2)
γ , deg	90	84.690(2)
V , Å ³	3658.8(7)	1468.4(2)
Z	4	2
$\mu(\text{MoK}\alpha)$, cm ⁻¹	1.420	1.638
$T_{\text{min}}/T_{\text{max}}$	0.7943/0.8480	0.7044/0.7353
ρ_{calcd} , g cm ⁻³	1.653	1.560
Collected reflections	5467	6043
Unique reflections	4239	4776
R_{int}	0.0701	0.0293
Restraints/parameters	38/506	0/365
GOOF	1.093	1.043
R_1, wR_2 ($I \geq 2\sigma(I)$)	0.0757, 0.1813	0.0403, 0.0887
R_1, wR_2 (all data)	0.1015, 0.1928	0.0566, 0.0975
$\Delta\rho_{\text{max}}/\Delta\rho_{\text{min}}$, e Å ⁻³	1.279/-0.748	0.997/-0.575

chelate rings Co(1)–N(5)–Co(2)–N(8) and Co(1)–N(11)–Co(3)–N(14). The coordinate bond lengths in the complex are comparable to those observed in the azido-bridged cobalt(III) complexes with Schiff bases [34–36]. The azido ligands are nearly linear, with NNN angles of 173.9(15)°–179.0(14)°.

The molecular structure of complex **II** is shown in Fig. 1b. The complex is a phenolate-bridged dinuclear copper(II) species. Molecule of the complex possesses a crystallographic inversion center symmetry, with the Cu...Cu distances of 3.13 Å. Each Cu atom is penta-coordinated with a square pyramidal geometry, the apical position being occupied by one phenolate O atom. The basal plane is defined by three donor atoms of the Schiff base ligand and one thiocyanate N atom. The distortion of the square pyramidal coordination can be observed by the bond angles among the apical and basal donor atoms, varying from 84.8(1)° to 103.2(1)° for Cu(1) and from 86.7(1)° to 102.8(1)° for Cu(2). The distortion is mainly arising from the strain created by the four-membered chelate rings Cu(1)–

O(1)–Cu(1A)–O(1A) and Cu(2)–O(2)–Cu(2A)–O(2A) (symmetry code for A: $-x, -y, 1-z$). The coordinate bond lengths in the complex are comparable to those observed in the phenoalte-bridged copper(II) complexes with Schiff bases [37–39].

The medium and broad absorption centered at 3506 cm⁻¹ in the spectrum of complex **I** substantiates the presence of O–H groups. The intense bands indicative of the azide vibrations of **I** are observed at 2057 and 2019 cm⁻¹, indicating there exist two different azide groups [40]. The intense band indicative of the thiocyanate vibrations of **II** is observed at 2098 cm⁻¹ [41]. The strong absorption bands at 1636 cm⁻¹ for complex **I** and 1632 cm⁻¹ for complex **II** are assigned to the azomethine groups, $\nu(\text{C}=\text{N})$ [42].

The electronic spectra of complexes **I** and **II** measured in methanol are shown in Fig. 2. The intense bands observed at about 230–280 nm for the complexes are assigned to intraligand $\pi-\pi^*$ transitions. The complexes displayed bands centered in the range

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

Bond	<i>d</i> , Å	Bond	<i>d</i> , Å
I			
Co(1)–O(4)	2.067(9)	Co(1)–O(3)	2.086(9)
Co(1)–N(14)	2.096(12)	Co(1)–N(8)	2.123(10)
Co(1)–N(11)	2.219(10)	Co(1)–N(5)	2.223(10)
Co(2)–N(1)	1.878(11)	Co(2)–O(1)	1.905(9)
Co(2)–N(17)	1.946(11)	Co(2)–N(8)	1.995(10)
Co(2)–N(5)	2.022(11)	Co(2)–N(2)	2.040(11)
Co(3)–N(3)	1.885(12)	Co(3)–O(2)	1.901(7)
Co(3)–N(20)	1.929(13)	Co(3)–N(14)	1.985(10)
Co(3)–N(11)	1.989(10)	Co(3)–N(4)	2.028(9)
II			
Cu(1)–N(5)	1.959(3)	Cu(1)–O(1)	1.9659(19)
Cu(1)–N(1)	1.987(2)	Cu(1)–N(2)	2.098(2)
Cu(1)–O(1A)	2.266(2)	Cu(2)–N(6)	1.955(3)
Cu(2)–O(2)	1.963(2)	Cu(2)–N(3)	1.983(2)
Cu(2)–N(4)	2.092(2)	Cu(2)–O(2A)	2.328(2)
Angle	ω , deg	Angle	ω , deg
I			
O(4)Co(1)O(3)	86.5(3)	O(4)Co(1)N(14)	170.2(4)
O(3)Co(1)N(14)	87.8(4)	O(4)Co(1)N(8)	88.7(4)
O(3)Co(1)N(8)	170.1(4)	N(14)Co(1)N(8)	98.1(3)
O(4)Co(1)N(11)	99.6(4)	O(3)Co(1)N(11)	85.1(4)
N(14)Co(1)N(11)	72.0(4)	N(8)Co(1)N(11)	104.3(4)
O(4)Co(1)N(5)	83.6(4)	O(3)Co(1)N(5)	98.0(4)
N(14)Co(1)N(5)	105.0(4)	N(8)Co(1)N(5)	72.8(4)
N(11)Co(1)N(5)	175.7(3)	N(1)Co(2)O(1)	93.3(4)
N(1)Co(2)N(17)	91.3(6)	O(1)Co(2)N(17)	90.6(5)
N(1)Co(2)N(8)	176.3(5)	O(1)Co(2)N(8)	87.8(4)
N(17)Co(2)N(8)	92.2(5)	N(1)Co(2)N(5)	96.5(5)
O(1)Co(2)N(5)	87.6(4)	N(17)Co(2)N(5)	172.0(5)
N(8)Co(2)N(5)	79.9(4)	N(1)Co(2)N(2)	87.1(5)
O(1)Co(2)N(2)	179.1(4)	N(17)Co(2)N(2)	88.6(5)
N(8)Co(2)N(2)	91.8(4)	N(5)Co(2)N(2)	93.2(5)
N(3)Co(3)O(2)	92.8(4)	N(3)Co(3)N(20)	90.8(5)
O(2)Co(3)N(20)	90.0(5)	N(3)Co(3)N(14)	176.4(5)
O(2)Co(3)N(14)	86.7(4)	N(20)Co(3)N(14)	92.7(5)
N(3)Co(3)N(11)	97.0(5)	O(2)Co(3)N(11)	88.9(4)
N(20)Co(3)N(11)	172.1(5)	N(14)Co(3)N(11)	79.4(4)
N(3)Co(3)N(4)	86.8(5)	O(2)Co(3)N(4)	176.6(4)
N(20)Co(3)N(4)	86.6(5)	N(14)Co(3)N(4)	93.9(4)
N(11)Co(3)N(4)	94.5(4)		
II			
N(5)Cu(1)O(1)	86.20(10)	N(5)Cu(1)N(1)	162.81(12)
O(1)Cu(1)N(1)	88.28(9)	N(5)Cu(1)N(2)	90.50(11)
O(1)Cu(1)N(2)	172.97(9)	N(1)Cu(1)N(2)	93.12(10)
N(5)Cu(1)O(1A)	103.22(11)	O(1)Cu(1)O(1A)	84.84(8)
N(1)Cu(1)O(1A)	92.46(9)	N(2)Cu(1)O(1A)	101.96(9)
N(6)Cu(2)O(2)	87.13(11)	N(6)Cu(2)N(3)	168.33(12)
O(2)Cu(2)N(3)	88.05(9)	N(6)Cu(2)N(4)	90.95(11)
O(2)Cu(2)N(4)	170.48(9)	N(3)Cu(2)N(4)	92.06(10)
N(6)Cu(2)O(2A)	100.03(11)	O(2)Cu(2)O(2A)	86.70(8)
N(3)Cu(2)O(2A)	90.29(9)	N(4)Cu(2)O(2A)	102.82(9)

* Symmetry code: (A) $-x, -y, 1 - z$.

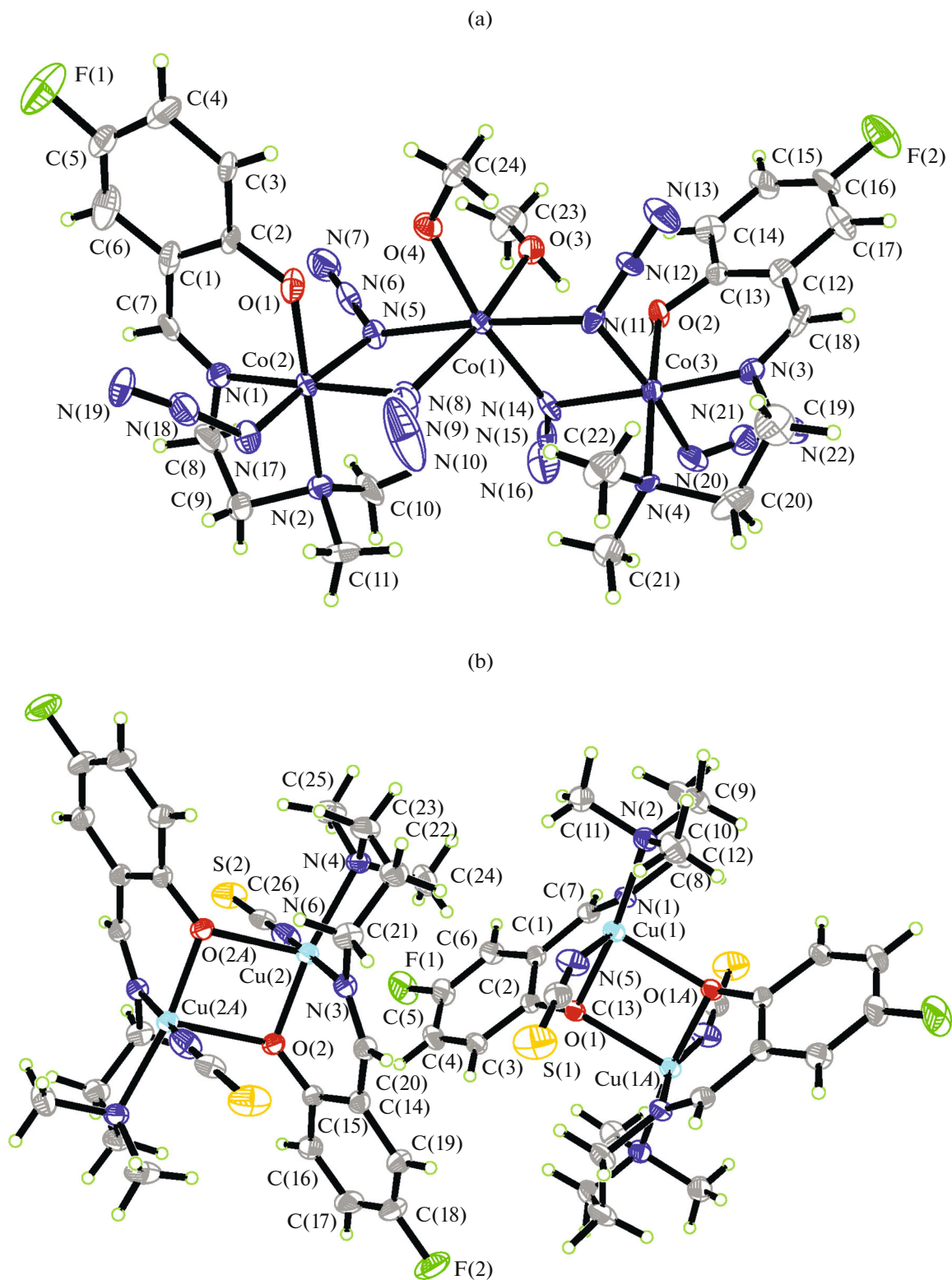


Fig. 1. A perspective view of the molecular structure of complexes I (a) and II (b) with the atom labeling scheme. Thermal ellipsoids are drawn at the 30% probability level. The carbon hydrogens are omitted for clarity. Atoms labelled with the suffix *A* or unlabelled are at the symmetry position $-x, -y, 1 - z$.

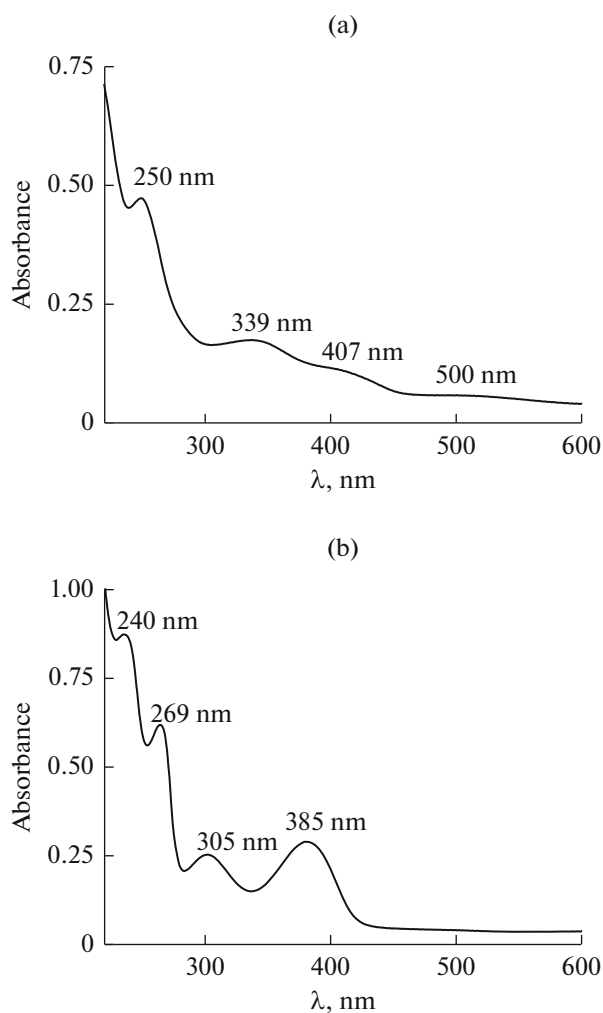


Fig. 2. UV spectrum of complexes I (a) and II (b).

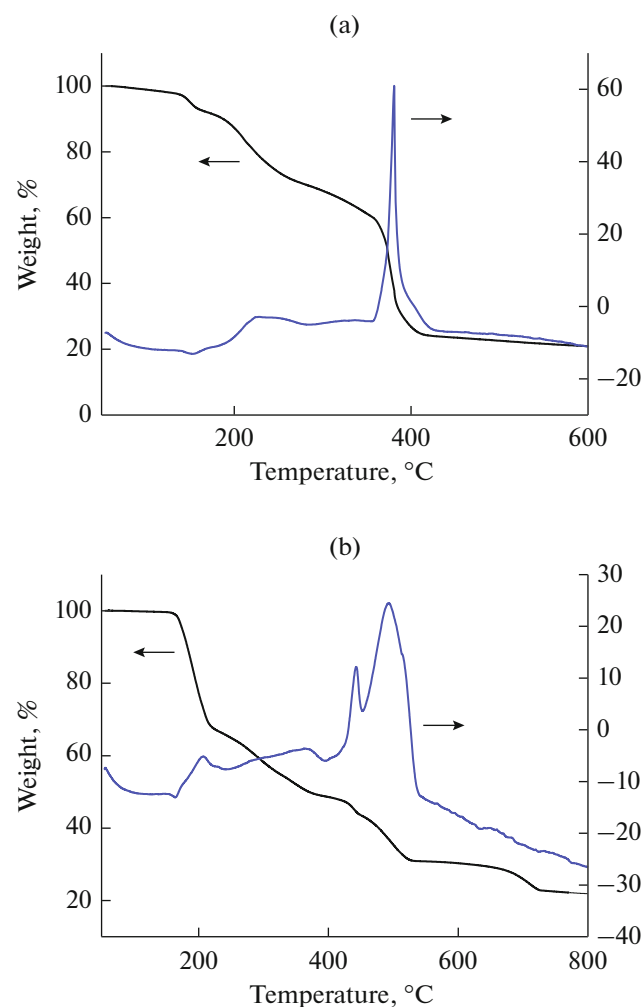


Fig. 3. DT-TG curves of complexes I (a) and II (b).

300–350 nm, which can be assigned to the $n-\pi^*$ transition. The charge transfer LMCT bands are located in the range of 380–500 nm.

Differential thermal (DT) and thermal gravimetric (TG) analyses were conducted to examine the stability of the complexes. For complex I (Fig. 3a), it decomposed from 139° to 425°C. The observed weight loss of 75.8% is close to the calculated value of 75.3% with

CoO as the final product. For complex II (Fig. 3b), it decomposed from 160° to 725°C. The observed weight loss of 77.3% is close to the calculated value of 77.0% with CuO as the final product.

The antibacterial results are summarized in Table 3. The Schiff bases show strong activity against *S. aureus*, and medium activity against *E. coli* and *C. parapsilosis*. The cobalt and copper complexes show

Table 3. Antibacterial property of the Schiff bases and complexes I and II

Compound	<i>S. aureus</i>		<i>E. coli</i>		<i>C. parapsilosis</i>	
	IC ₅₀ *	MIC*	IC ₅₀	MIC	IC ₅₀	MIC
HL ¹	0.56	>2.5	1.35	>2.5	3.72	>2.5
HL ²	0.71	>2.5	1.63	>2.5	2.81	>2.5
I	0.32	0.54	0.71	1.03	1.12	>2.5
II	0.17	0.28	0.45	0.65	0.72	1.81

* mmol L⁻¹.

more effective activity against *S. aureus*, *E. coli* and *C. parapsilosis* than the Schiff bases. It is obvious that the copper complex is more effective than the cobalt complex for the bacteria. The copper complex has the most activity against *S. aureus* with IC₅₀ and MIC values of 0.17 and 0.28 mmol L⁻¹, which deserve further study.

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