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Synthesis, Characterization, Crystal Structures, and Antimicrobial Activity of Cobalt(II) and Iron(III) Complexes Derived from N'-(2-Hydroxybenzylidene)-3-Methylbenzohydrazide[1]

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Abstract—A new cobalt(II,III) complex, $[Co^{III}L_2]_2[Co^{II}_2(HL)_2(OH_2)_2(CH_3OH)_2] \cdot 2H_2O$ (**I**) and a new iron(III) complex, $[Fe^{III}(HL)_2](NO_3)$ (**II**), where L^{2-} and HL^- are the dianionic and monoanionic form of N'-(2-hydroxybenzylidene)-3-methylbenzohydrazide, respectively, have been prepared and characterized by elemental analyses, infrared and UV-Vis spectroscopy and single-cyrstal X-ray diffraction (CIF files CCDC nos. 1417971 (**I**), 1417979 (**II**)). Complex **I** crystallizes in the monoclinic space group $P2_1/n$ with unit cell dimensions $a = 16.1665(9)$, $b = 14.5692(8)$, $c = 19.086(1)$ Å, $\beta = 96.347(1)°$, $V = 4467.9(4)$ Å^3, $Z = 2$, $R_1 = 0.0521$, and $wR_2 = 0.1411$. Complex **II** crystallizes in the orthorhombic space group *Pbcn* with unit cell dimensions $a = 12.475(1)$, $b = 12.202(1)$, $c = 18.859(2)$ Å, $V = 2870.8(4)$ Å^3, $Z = 4$, $R_1 = 0.0796$, and $wR_2 = 0.1981$. The metal atoms in the complexes are in octahedral coordination. Crystals of the complexes are stabilized by hydrogen bonds. The efficiency of the aroylhydrazone and the two complexes was evaluated against *B. subtilis*, *S. aureus*, *E. coli*, *P. fluorescence*, *C. albicans* and *A. niger*, with the complexes demonstrating enhanced activity relatively to the free ligand.

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INTRODUCTION

Schiff bases and their complexes have received considerable attention for their importance in the field of coordination chemistry related to catalysis and enzymatic reactions, magnetism and molecular architectures [1–5]. Aroylhydrazone is a special type of Schiff base, which possess interesting biological activities such as antimicrobial and antitumor [6–12]. In recent years, the biological activities of metal complexes derived from aroylhydrazones have received considerable attention [13–20]. We have reported the antimicrobial activities of some aroylhydrazone compounds [21], and the urease inhibitory activities of some oxovanadium complexes with aroylhydrazone ligands [22–25]. In the present work, the synthesis, structural characterization and antimicrobial activity of a new cobalt(II/III) complex, $[Co^{III}L_2]_2[Co^{II}_2(HL)_2(OH_2)_2(CH_3OH)_2] \cdot 2H_2O$ (**I**) and a new iron(III) complex, $[Fe^{III}(HL)_2](NO_3)$ (**II**), where L^{2-} and HL^- are the dianionic and monoanionic form of N'-(2-hydroxybenzylidene)-3-methylbenzohydrazide (H_2L), respectively, are presented.

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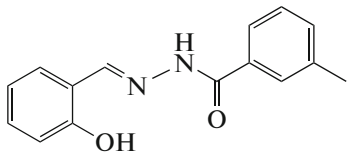


(H_2L)

EXPERIMENTAL

Materials and measurements. Commercially available salicylaldehyde and 3-methylbenzohydrazide were purchased from Aldrich and used without further purification. Other solvents and reagents were made in China and used as received. The aroylhydrazone compound was prepared according to the literature procedure [26]. C, H, and N elemental analyses were performed with a Perkin-Elmer elemental analyser. Infrared spectra were recorded on a Nicolet AVATAR 360 spectrometer as KBr pellets in the 4000–400 cm^{-1} region. UV-Vis spectra were recorded on a Lambda 900 spectrometer.

Synthesis of I. A MeOH solution (10 mL) of $CoCl_2$ (1.0 mmol, 0.234 g) was added to a MeOH solution (20 mL) of H_2L (1.0 mmol, 0.254 g) with stirring. The

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

Parameter	Value	
	I	**II**
Fw	1887.4	624.4
Crystal shape/color	Block/red	Block/dark
Crystal size, mm	0.31 × 0.28 × 0.26	0.23 × 0.20 × 0.20
Crystal system	Monoclinic	Orthorhombic
Space group	$P2_1/n$	*Pbcn*
a, Å	16.1665(9)	12.475(1)
b, Å	14.5692(8)	12.202(1)
c, Å	19.086(1)	18.859(2)
β, deg	96.347(1)	
V, Å^3	4467.9(4)	2870.8(4)
Z	2	4
μ(MoK_α), mm^{-1}	0.804	0.582
ρ_{calcd}, g cm^{-3}	1.400	1.445
Reflections/parameters	8316/585	2568/200
Restraints	1	1
Observed reflections ($I \geqslant 2\sigma(I)$)	5812	1579
F(000)	1948	1292
T_{min}/T_{max}	0.7886/0.8181	0.8778/0.8925
Goodness of fit on F^2	1.039	1.043
R_1, wR_2 ($I \geqslant 2\sigma(I)$)*	0.0521, 0.1411	0.0796, 0.1981
R_1, wR_2 (all data)*	0.0809, 0.1584	0.1352, 0.2372
Largest peak and deepest hole, *e* Å^{-3})	0.818, –0.381	1.495, –0.415

* $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$, $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{1/2}$.

mixture was stirred for 30 min to give a clear red solution. The resulting solution was allowed to stand in air for a few days. Red block-shaped crystals suitable for X-ray single crystal analysis were formed at the bottom of the vessel. The isolated product was washed three times with cold MeOH and dried in a vacuum over anhydrous $CaCl_2$. The yield was 17.2 mg (36%).

IR data (KBr; ν, cm^{-1}): 3522 w, 3189 w, 1603 s, 1545 m, 1517 s, 1443 w, 1379 m, 1334 m, 1290 m, 1220 w, 1195 w, 1147 w, 1094 w, 1018 w, 949 w, 898 m, 798 w, 743 m, 725 m, 686 w, 644 w, 607 w, 521 w, 493 w, 431 w. UV-Vis data in acetonitrile (λ_{max}, nm): 268, 325, 389, 405.

For $C_{92}H_{90}N_{12}O_{18}Co_4$

anal. calcd. %:	C, 58.54;	H, 4.81;	N, 8.90.
Found, %:	C, 58.40;	H, 4.69;	N, 8.82.

Synthesis of II. A MeOH solution (10 mL) of $Fe(NO_3)_3$ (1.0 mmol, 0.404 g) was added to a MeOH solution (20 mL) of H_2L (1.0 mmol, 0.254 g) with stirring. The mixture was stirred for 30 min to give a clear brown solution. The resulting solution was allowed to stand in air for a few days. Dark block-shaped crystals suitable for X-ray single crystal analysis were formed at the bottom of the vessel. The isolated product was washed three times with cold MeOH and dried in a vacuum over anhydrous $CaCl_2$. The yield was 19.3 mg (62%).

IR data (KBr; ν, cm^{-1}): 3195 w, 1608 s, 1536 w, 1432 s, 1393 m, 1357 w, 1273 w, 1144 s, 1083 s, 996 m, 954 m, 856 m, 758 w, 727 w, 549 m, 470 w. UV-Vis data in acetonitrile (λ_{max}, nm): 290, 360, 450.

For $C_{30}H_{26}N_5O_7Fe$

anal. calcd. %:	C, 57.60;	H, 4.19;	N, 11.20.
Found, %:	C, 57.81;	H, 4.27;	N, 11.08.

X-ray structure determination. Diffraction intensities for the complexes were collected at 298(2) K using

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

Bond	*d*, Å	Bond	*d*, Å
		I	
Co(1)–N(1)	1.881(3)	Co(1)–O(2)	1.890(3)
Co(1)–N(3)	1.871(3)	Co(1)–O(3)	1.871(3)
Co(1)–O(4)	1.891(3)	Co(1)–O(1)	1.897(3)
Co(2)–O(5)	2.021(2)	Co(2)–N(5)	2.051(3)
Co(2)–O(7)	2.071(3)	Co(2)–O(5*A*)	2.098(2)
Co(2)–O(8)	2.101(3)	Co(2)–O(6)	2.111(3)
		II	
Fe(1)–O(1)	1.897(4)	Fe(1)–O(2)	2.061(4)
Fe(1)–N(1)	2.113(4)		
Angle	ω, deg	Angle	ω, deg
		I	
N(3)Co(1)O(3)	95.5(1)	N(3)Co(1)N(1)	172.4(1)
O(3)Co(1)N(1)	89.6(1)	N(3)Co(1)O(2)	91.0(1)
O(3)Co(1)O(2)	89.5(1)	N(1)Co(1)O(2)	83.4(1)
N(3)Co(1)O(4)	83.4(1)	O(3)Co(1)O(4)	178.5(1)
N(1)Co(1)O(4)	91.6(1)	O(2)Co(1)O(4)	91.5(1)
N(3)Co(1)O(1)	90.6(1)	O(3)Co(1)O(1)	89.4(1)
N(1)Co(1)O(1)	95.0(1)	O(2)Co(1)O(1)	178.1(1)
O(4)Co(1)O(1)	89.5(1)	O(5)Co(2)N(5)	87.9(1)
O(5)Co(2)O(7)	94.0(1)	N(5)Co(2)O(7)	94.8(1)
O(5)Co(2)O(5*A*)	81.6(1)	N(5)Co(2)O(5*A*)	169.4(1)
O(7)Co(2)O(5*A*)	84.0(1)	O(5)Co(2)O(8)	93.6(1)
N(5)Co(2)O(8)	98.0(1)	O(7)Co(2)O(8)	165.3(1)
O(5*A*)Co(2)O(8)	84.7(1)	O(5)Co(2)O(6)	164.3(1)
N(5)Co(2)O(6)	76.6(1)	O(7)Co(2)O(6)	89.9(1)
O(5*A*)Co(2)O(6)	113.9(1)	O(8)Co(2)O(6)	86.2(1)
		II	
O(1)Fe(1)O(1*A*)	99.8(3)	O(1)Fe(1)O(2)	157.9(2)
O(1)Fe(1)O(2*A*)	91.5(2)	O(2)Fe(1)O(2*A*)	84.6(2)
O(1)Fe(1)N(1*A*)	102.9(2)	O(2)Fe(1)N(1*A*)	97.0(2)
O(1)Fe(1)N(1)	84.2(2)	O(2)Fe(1)N(1)	74.7(2)
N(1)Fe(1)N(1*A*)	169.1(2)		

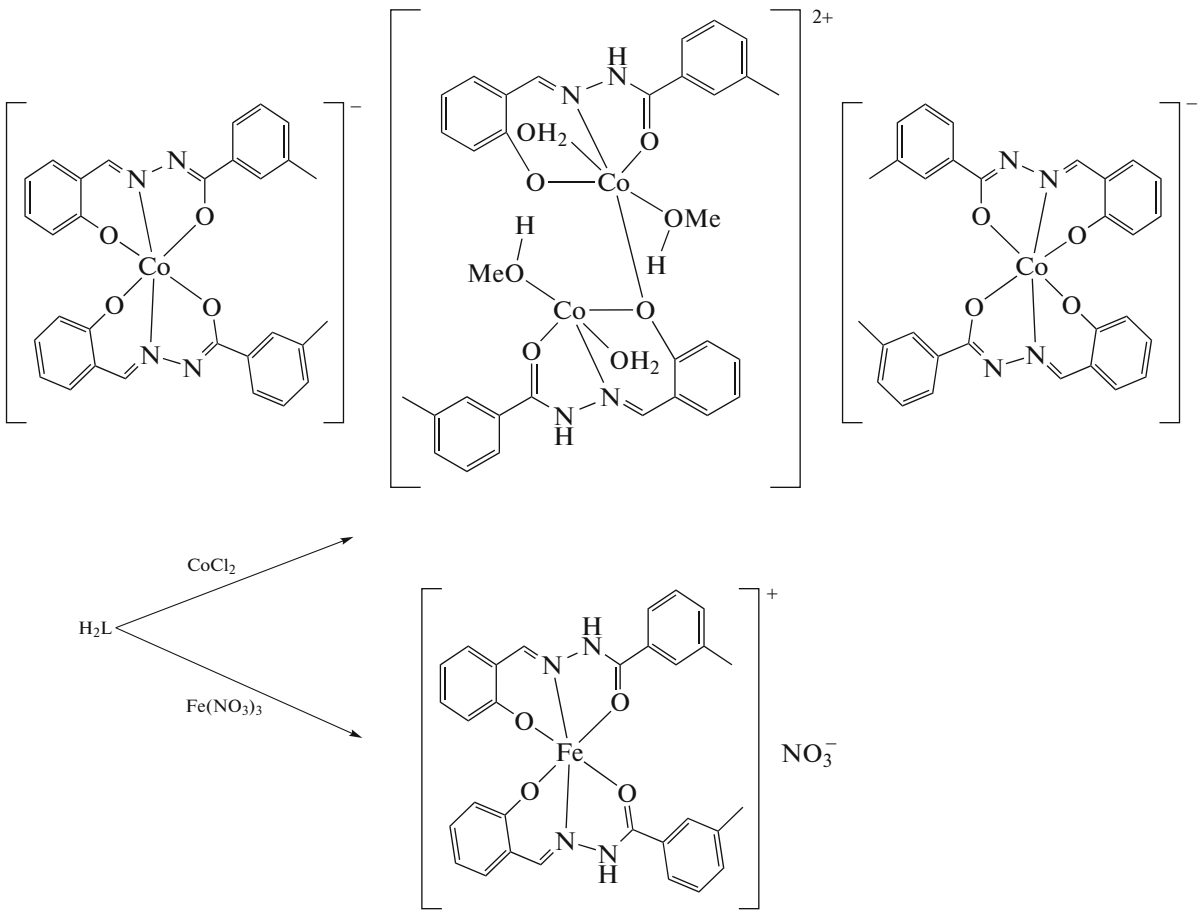
a Bruker SMART 1000 CCD area-detector with MoK_α radiation (λ = 0.71073 Å). The collected data were reduced using SAINT [27], and multi-scan absorption corrections were performed using SADABS [28]. The structures were solved by direct methods and refined against F^2 by full-matrix least-squares methods using SHELXTL [29]. All of the non-hydrogen atoms were refined anisotropically. The amino H atoms in both structures were located from difference Fourier maps and restrained isotropically. The remaining hydrogen atoms were placed in idealized positions and constrained to ride on their parent atoms. The isolated water molecules in **I** are disordered with occupancy of 0.5 each. The hydrogen atoms of the disordered water molecules are not added. Crystallographic data for the complexes are summarized in Table 1. Selected bond lengths and angles are given in Table 2. Supplementary material has been deposited with the Cambridge Crystallographic Data Centre (nos. 1417971 (**I**), 1417979 (**II**)); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

Antimicrobial assay. The antibacterial activities of the compounds were tested against *B. subtilis*,

S. aureus, *E. coli*, and *P. fluorescence* using MH (Mueller–Hinton) medium. The antifungal activities of the compounds were tested against *C. albicans* and *A. niger* using RPMI-1640 medium. The MIC (minimum inhibitory concentration) values of the tested compounds were determined by a colorimetric method using the dye MTT [30]. A stock solution of the aroylhydrazone compound (150 μM) in DMSO was prepared and graded quantities (75, 37.5, 18.8, 9.4, 4.7, 2.3, 1.2, 0.59 μM) of the tested compounds were incorporated in specified quantity of the corresponding sterilized liquid medium. A specified quantity of the medium containing the compound was poured into microtitration plates. Suspension of the microorganism was prepared to contain approximately 10^5 cfu/mL and applied to microtitration plates with serially diluted compounds in DMSO to be tested and incubated at 37°C for 24 and 48 h for bacterial and fungi, respectively. Then the MIC values were visually determined on each of the microtitration plates, 50 μL of PBS (phosphate buffered saline 0.01 M, pH 7.4) containing 2 mg of MTT/mL 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 M 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 550 nm.

RESULTS AND DISCUSSION

Complexes **I** and **II** were prepared by the reaction of the aroylhydrazone with cobalt chloride and iron nitrate, respectively:



For complex **I**, there form two different cobalt complexes in the crystal. In the mononuclear complex, the Co atom is in +3 oxidation state, which was oxidized by oxygen of air. In the dinuclear complex, the Co atom is in +2 oxidation state. For complex **II**, the Fe atom is in +3 oxidation state. The crystals of the complexes are stable in air at room temperature, soluble in common polar organic solvents, such as DMSO, DMF, MeOH, EtOH, MeCN and Me_2CO, and insoluble in water and Et_2O.

The medium and sharp absorption at about 3190 cm^{-1} can be assigned to ν(N–H). Strong absorptions centered at 1603 cm^{-1} for complex **I** and 1608 cm^{-1} for complex **II** are assigned to azomethine, ν(C=N) [31]. The bands at 1094 cm^{-1} for **I** and

Table 3. Geometric parameters of hydrogen bonds for complexes **I**, **II***

D–H···A	Distance, Å			D–H···A
	D–H	H···A	D···A	
		I		
N(6)–H(6)···O(1)	0.90(1)	1.97(1)	2.872(4)	174(4)
O(8)–H(8*A*)···O(9)^{i}	0.93	2.62	3.338(10)	134
O(8)–H(8*A*)···O(7)^{ii}	0.93	2.24	3.064(5)	147
O(7)–H(7*B*)···O(8)^{ii}	0.85	2.40	3.064(5)	136
O(7)–H(7*A*)···N(1)^{iii}	0.85	2.69	3.338(4)	135
		II		
N(2)–H(2)···O(3)	0.90(1)	1.86(2)	2.742(6)	170(7)

* Symmetry codes: ^{i} $1-x, 1-y, 1-z$; ^{ii} $1-x, -y, 1-z$; ^{iii} $3/2-x, -1/2+y, 3/2-z$.

1083 cm^{-1} for **II** are assigned to C–O absorption. The aroylhydrazone ligand coordination to the metal atoms is also substantiated by weak bands at low wavenumbers.

The electronic spectra of the complexes **I** and **II** in acetonitrile show two strong absorption bands centered at 268 and 290 nm, respectively. These bands are clearly charge-transfer bands [32]. The bands centered at 325 and 389 nm for **I**, and 360 nm for **II** are assigned to the $n-\pi^*$ transition of the azomethine group. The visible spectra of the complexes display single broad bands at 405 nm for **I** and 450 nm for **II**, which can be explained by *d–d* transition.

From X-ray diffraction analysis of the free aroylhydrazone [26], the ligand in its free form presents in the *keto* tautomeric form with the structural conformation of the molecule being *E* with respect to the imine double bond. During the process of complexation the aroylhydrazone molecule can coordinate to the metal atoms through either the keto form or the enol form.

The molecular structure of **I** is shown in Fig. 1a. The asymmetric unit of the compound contains two mononuclear cobalt(III) complex anions and one centrosymmetric phenolate bridged dinuclear cobalt(II) complex canion. In the mononuclear complex anions, the Co atoms are in +3 oxidation state, and in the dinuclear complex cation, the Co atoms are in +2 oxidation state. This can be verified by the coordinate bond lengths. The bond lengths of Co(1)–N(1) 1.88 and Co(1)–N(3) 1.87 Å in the mononuclear complex anions are much shorter than Co(2)–N(5) 2.05 Å in the dinuclear complex canion. The same phenomenon can be observed from the Co–O bonds (Table 2). For the mononuclear complex molecule, the Co atom is coordinated by two phenolate O, two imino N and two enolate O atoms from two dianionic aroylhydrazone ligands, forming an octahedral geometry. The Co–O and Co–N bond lengths are comparable to those observed in cobalt(III) complexes with hydrazone ligands [33, 34]. For the dinuclear complex molecule, the Co atom is also in octahedral coordination, with the equatorial plane defined by the phenolate O, imino N and carbonyl O atom of a monoanionic aroylhydrazone ligand, and the phenolate O of another aroylhydrazone ligand, and with the two axial positions occupied by one water and one methanol molecules. The phenolate O atom of the dinuclear complex molecule acts as a bridging group, which coordinates to two Co atoms, with the Co···Co separation of 3.118(2) Å. The Co–O and Co–N bond lengths in the dinuclear complex molecule are comparable to those observed in cobalt(II) complexes with hydrazone ligands [35, 36]. In the crystal structure of the complex (Fig. 1b), the components are linked through N–H···O, O–H···O and O–H···N hydrogen bonds (Table 3) to form a 3D network.

The molecular structure of **II** is shown in Fig. 2a. The asymmetric unit of the compound contains a mononuclear cobalt(III) complex cation and a nitrate anion. The mononuclear complex molecule posseses a crystallographic two-fold rotation axis symmetry. The Fe atom is in +3 oxidation state, and is coordinated by two phenolate O, two imino N and two carbonyl O atoms from two monoanionic aroylhydrazone ligands, forming an octahedral geometry. The Fe–O and Fe–N bond lengths are comparable to those observed in iron(III) complexes with hydrazone ligands [37, 38]. In the crystal structure of the complex (Fig. 2b), the components are linked through N–H···O hydrogen bonds (Table 3), to form 1D chains along the *x* axis direction.

The compounds were screened for antibacterial activity against two Gram (+) bacterial strains (*Bacillus subtilis* and *Staphylococcus aureus*) and two Gram (–) bacterial strains (*Escherichia coli* and *Pseudomonas fluorescence*) by MTT method. The MIC values of the compounds against four bacteria are listed in Table 4. Kanamycin and Penicillin G were used as the standard materials. The free aroylhydrazone compound has weak activity against *S. aureus*, and no

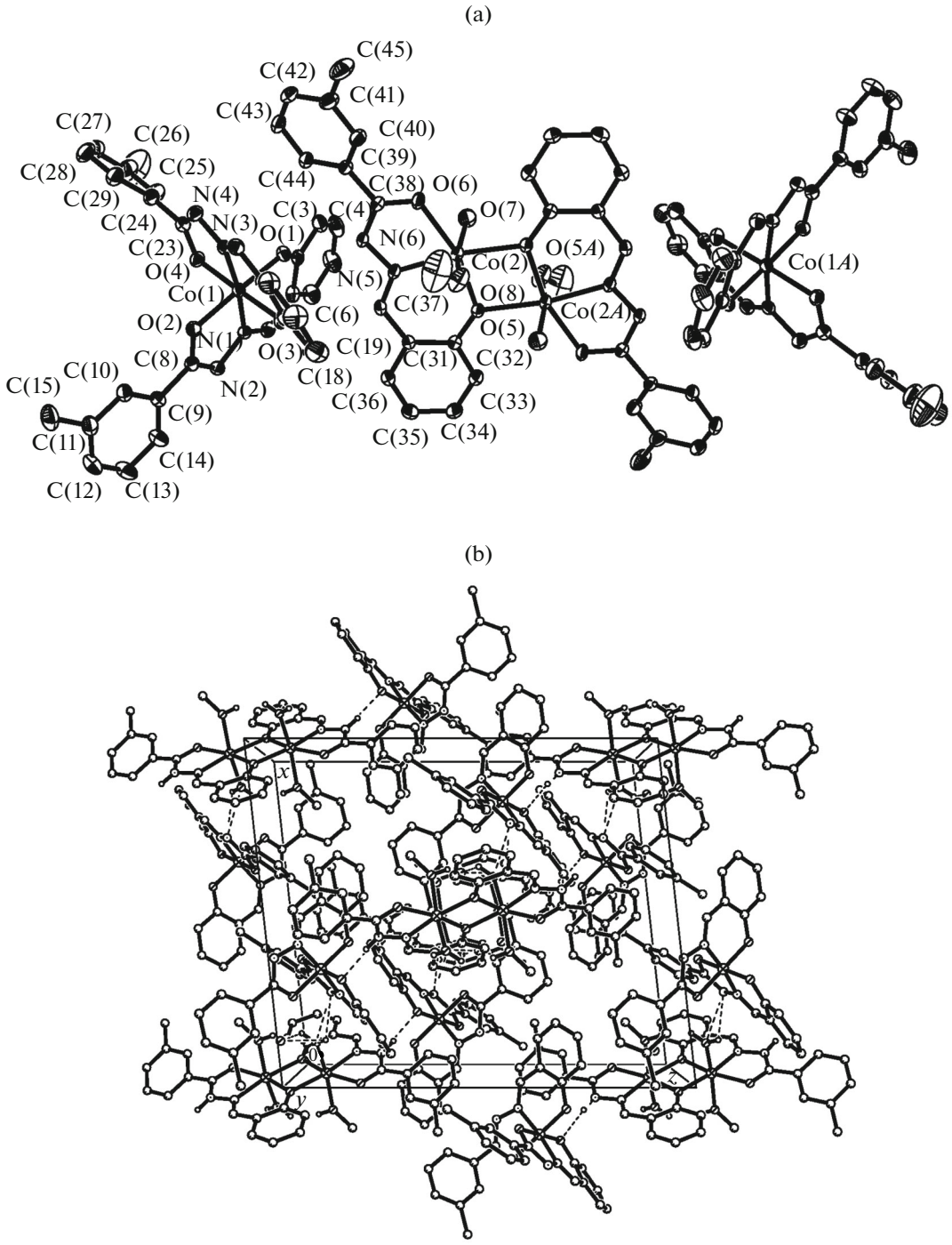


Fig. 1. Molecular structure of **I** at 30% probability displacement (atoms labeled with the suffix *A* or unlabeled are at the symmetry position $1-x, -y, 1-z$) (a); the 3D network structure of **I**, viewed along the *y* axis (hydrogen bonds are drawn as dashed lines) (b).

activity against the remaining bacterial strains. Complex **I** has strong activity against *B. subtilis* and *S. aureus*, and weak activity against *E. coli* and *P. fluorescence*. Complex **II** has medium activity against *B. subtilis*, weak activity against *S. aureus*, and no activity against *E. coli* and *P. fluorescence*. In general, the complexes have stronger activity against *B. subtilis* and *S. aureus* when compared to H_2L. Complex **I** has stronger activity against all the bacterial strains than complex **II**.

The antifungal activities of the compounds were also evaluated against two fungal strains (*Candida albicans* and *Aspergillus niger*) by MTT method. Keto-

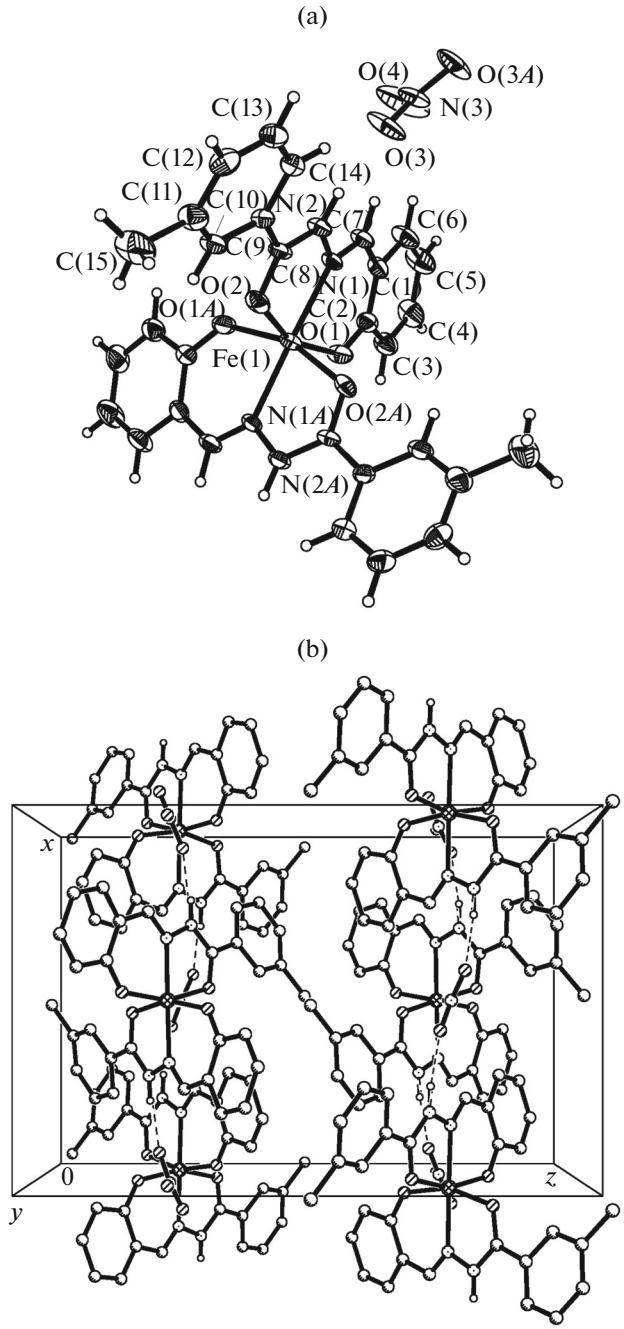


Fig. 2. Molecular structure of **II** at 30% probability displacement (atoms labeled with the suffix *A* or unlabeled are at the symmetry position $1 - x, y, 1/2 - z$) (a); the 1D chain structure of **II**, viewed along the *y* axis (hydrogen bonds are drawn as dashed lines) (b).

Table 4. The MIC values (μM) of the compounds

Tested material	*B. subtilis*	*S. aureus*	*E. coli*	*P. fluorescence*	*C. albicans*	*A. niger*
I	4.7	9.4	75	37.5	18.8	>150
II	18.8	37.5	>150	>150	>150	>150
H_2L	>150	75	>150	>150	>150	>150
Kanamycin	0.59	2.3	4.7	4.7	>150	>150
Penicillin G	2.3	4.7	>150	>150	>150	>150
Ketoconazole	>150	>150	>150	>150	4.7	18.8

conazole was used as a reference mateiral. As a result, complex **I** has medium activity against *C. albicans*. Complex **II** has no activity against both fungal strains.

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