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Synthesis, Crystal Structures, and Antibacterial Activity of Manganese(III) Complexes with Schiff Bases

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Abstract—Two new manganese(III) complexes, $[MnL^1(EtOH)(Acac)]$ (**I**) and $[MnL^2(DMF)(Esal)] \cdot H_2O$ (**II**), where L^1 and L^2 are the dianionic form of 2-[(2-hydroxyphenylimino)methyl]-6-methoxyphenol (H_2L^1) and 4-chloro-2-[(3-ethoxy-2-hydroxybenzylidene)amino]phenol (H_2L^2), respectively, Acac is acetylacetone, Esal is 3-ethoxysalicylaldehyde, were prepared and characterized by IR and UV-Vis spectra, as well as single crystal X-ray diffraction (CIF files CCDC nos. 1849854 (**I**) and 1849855 (**II**)). Complex **I** crystallizes as the hexagonal space group $P\bar{3}$ with unit cell dimensions $a = b = 20.4482(9)$, $c = 8.6952(7)$ Å, $V = 3148.6(3)$ Å^3, $Z = 6$, $R_1 = 0.0375$, $wR_2 = 0.0957$, GOOF = 1.050. Complex **II** crystallizes as the triclinic space group $P\bar{1}$ with unit cell dimensions $a = 8.1602(12)$, $b = 11.5960(15)$, $c = 15.3859(13)$ Å, $\alpha = 78.873(2)°$, $\beta = 83.766(2)°$, $\gamma = 84.964(2)°$, $V = 1416.7(3)$ Å^3, $Z = 2$, $R_1 = 0.0733$, $wR_2 = 0.1795$, GOOF = 1.029. X-ray analyses indicate that the complexes are manganese(III) species, with the Mn atoms in octahedral coordination. The Schiff bases and the complexes were evaluated for their antibacterial (*Bacillus subtilis*, *Staphylococcus aureus*, *Escherichia coli*, and *Pseudomonas fluorescence*) activities.

Keywords: Schiff base, manganese complex, mononuclear complex, crystal structure, antibacterial activity

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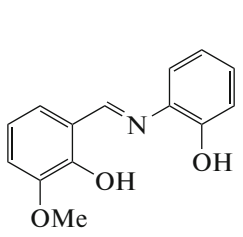
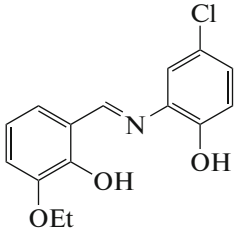
INTRODUCTION

Schiff bases are a kind of biological active compound, which can be prepared by the condensation reaction of carbonyl-containing compounds with various amines. The compounds have attracted considerable attention for their wide range of biological activities [1–7]. It was reported that compounds bearing electron-withdrawing groups such as halide can improve their antimicrobial activities [8, 9]. Series of fluoro, chloro, bromo, and iodo-substituted compounds, and found that they have significant antimicrobial activities were described in [10]. Manganese complexes with Schiff bases have been reported to have interesting antibacterial activities [11–14]. In the present work, two new manganese(III) complexes, $[MnL^1(EtOH)(Acac)]$ (**I**) and $[MnL^2(DMF)(Esal)] \cdot H_2O$ (**II**), where L^1 and L^2 are the dianionic form of 2-[(2-hydroxyphenylimino)methyl]-6-methoxyphenol (H_2L^1) and 4-chloro-2-[(3-ethoxy-2-hydroxybenzylidene)amino]phenol (H_2L^2), respectively, Acac is acetylacetone, Esal is 3-ethoxysalicylaldehyde, are reported.



(H_2L^1)



(H_2L^2)

EXPERIMENTAL

Materials and methods. 3-Methoxysalicylaldehyde, 3-ethoxysalicylaldehyde, 2-aminophenol and $MoO_2(Acac)_2$ were purchased from Sigma-Aldrich and used as received. All other reagents were of analytical reagent grade. Elemental analyses of C, H and N were carried out in a Perkin-Elmer automated model 2400 Series II CHNS/O analyzer. FT-IR spectra were obtained on a Perkin-Elmer 377 FT-IR spectrometer with samples prepared as KBr pellets. UV-Vis spectra were obtained on a Lambda 900 spectrometer. ^{1}H NMR and ^{13}C NMR data was recorded on a Bruker 500 MHz spectrometer. X-ray diffraction was carried out on a Bruker APEX II CCD diffractometer.

Synthesis of H_2L^1. 3-Methoxysalicylaldehyde (0.01 mol, 1.52 g) and 2-aminophenol (0.01 mol, 1.10 g) were dissolved in ethanol (50 mL). The mixture was stirred for 30 min to give red solution. Then, the solvent was removed by distillation. The red solid was recrystallized from ethanol to give the Schiff base H_2L^1. The yield was 2.12 g (87%).

IR data (ν, cm^{-1}): 3432 (O–H), 1628 (C=N), 1277 (Ar–O). UV-Vis data (CH_3OH; λ_{max}, nm): 267, 346, 451. ^{1}H NMR (500 MHz; DMSO; δ, ppm): 14.02 (s., 1H, OH), 9.74 (s., 1H, OH), 8.95 (s., 1H, CH=N), 7.37 (d., 1H, ArH), 7.20–7.07 (m., 3H, ArH), 6.98–6.84 (m., 3H, ArH), 3.81 (s., 3H, CH_3). ^{13}C NMR (126 MHz; DMSO; δ, ppm): 161.54, 151.80, 150.95, 148.15, 134.47, 127.98, 123.78, 119.57, 119.48, 119.22, 117.89, 116.49, 115.22, 55.85.

For $C_{14}H_{13}NO_3$

Anal. calcd., %	C, 69.12	H, 5.39	N, 5.76
Found, %	C, 68.95	H, 5.50	N, 5.83

Synthesis of H_2L^2. 3-Ethoxysalicylaldehyde (0.01 mol, 1.66 g) and 2-amino-4-chlorophenol (0.01 mol, 1.45 g) were dissolved in ethanol (50 mL). The mixture was stirred for 30 min to give red solution. Then, the solvent was removed by distillation. The red solid was recrystallized from ethanol to give the Schiff base H_2L^2. The yield was 2.52 g (86%).

IR data (ν, cm^{-1}): 3439 (O–H), 1625 (C=N), 1289 (Ar–O). UV-Vis data (CH_3OH; λ_{max}, nm): 265, 300, 345, 450. ^{1}H NMR (500 MHz; DMSO; δ, ppm): 13.70 (s., 1H, OH), 10.01 (s., 1H, OH), 8.98 (s., 1H, CH=N), 7.49 (s., 1H, ArH), 7.20–7.15 (m., 2H, ArH), 7.09 (d., 1H, ArH), 6.97 (d., 1H, ArH), 6.86 (t., 1H, ArH), 4.07 (q., 2H, CH_2), 1.34 (t., 3H, CH_3). ^{13}C NMR (126 MHz; DMSO; δ, ppm): 162.96, 151.68, 150.11, 147.17, 135.84, 127.30, 124.13, 123.08, 119.33, 119.10, 118.18, 117.76, 116.99, 64.09, 14.77.

For $C_{15}H_{14}ClNO_3$

Anal. calcd., %	C, 61.76	H, 4.84	N, 4.80
Found, %	C, 61.57	H, 4.93	N, 4.71

Synthesis of complex I. H_2L^1 (0.01 mol, 2.43 g) and $Mn(Acac)_2$ (0.01 mol, 2.53 g) were mixed and dissolved in ethanol (50 mL). The mixture was stirred for 1 h to give brown solution. Single crystals of the complex, suitable for X-ray diffraction, were grown from the solution upon slowly evaporation for a few days. The yield was 2.03 g (45%).

IR data (ν, cm^{-1}): 3434 (O–H), 1658 (C=O), 1596 (C=N), 1257 (Ar–O). UV-Vis data (CH_3OH; λ_{max}, nm): 250, 430.

For $C_{21}H_{24}NO_6Mn$

Anal. calcd., %	C, 57.15	H, 5.48	N, 3.17
Found, %	C, 57.33	H, 5.57	N, 3.08

Synthesis of complex II. H_2L^2 (0.01 mol, 2.91 g) and $Mn(CH_3COO)_2 \cdot 4H_2O$ (0.01 mol, 2.45 g) were mixed and dissolved in ethanol (50 mL) and DMF (10 mL). The mixture was stirred for 1 h to give yellow solution. Single crystals of the complex, suitable for X-ray diffraction, were grown from the solution upon slowly evaporation for a few days. The yield was 2.33 g (38%).

IR data (ν, cm^{-1}): 3357 (O–H), 1655 (C=O), 1598 (C=N), 1261 (Ar–O). UV-Vis data (CH_3OH; λ_{max}, nm): 245, 435.

For $C_{27}H_{30}N_2O_8ClMn$

Anal. calcd., %	C, 53.96	H, 5.03	N, 4.66
Found, %	C, 54.21	H, 5.12	N, 4.57

X-ray crystallography. X-ray diffraction was carried out at a Bruker APEX II CCD area diffractometer equipped with MoK_α radiation (λ = 0.71073 Å). The collected data were reduced with SAINT [15], and multi-scan absorption correction was performed using SADABS [16]. The structures of the complexes were solved by direct method and refined against F^2 by full-matrix least-squares method using SHELXTL [17]. All of the non-hydrogen atoms were refined anisotropically. The ethanol hydrogen atom in complex **I** was located from an electronic density map and refined isotropically with O–H distance restrained to 0.85(1) Å. The remaining hydrogen atoms were placed in calculated positions and constrained to ride on their parent atoms. The crystallographic data and refinement parameters for the complexes are listed in Table 1. Selected bond lengths and angles are listed in Table 2.

Table 1. Crystallographic data and structure refinement for **I** and **II**

Parameter	Value	
	I	**II**
Formula weight	441.35	600.92
Crystal shape/color	Block/brown	Block/brown
T, K	298(2)	298(2)
Crystal dimensions, mm	0.26 × 0.23 × 0.20	0.20 × 0.18 × 0.15
Crystal system	Hexagonal	Triclinic
Space group	$P\bar{3}$	$P\bar{1}$
a, Å	20.4482(9)	8.1602(12)
b, Å	20.4482(9)	11.5960(15)
c, Å	8.6952(7)	15.3859(13)
α, deg	90	78.873(2)
β, deg	90	83.766(2)
γ, deg	120	84.964(2)
V, Å^3	3148.6(3)	1416.7(3)
Z	6	2
ρ_{calcd}, g cm^{-3}	1.397	1.409
μ (MoK_α), mm^{-1}	0.665	0.611
F(000)	1380	624
Measured reflections	18857	8300
Unique reflections (R_{int})	3909 (0.0415)	5244 (0.0468)
Observed reflections ($I \geq 2\sigma(I)$)	3001	2957
Min and max transmission	0.8460 and 0.8784	0.8876 and 0.9140
Parameters	267	359
Restraints	1	0
Goodness of fit on F^2	1.050	1.029
R_1, wR_2 ($I \geq 2\sigma(I)$)*	0.0375, 0.0957	0.0733, 0.1795
R_1, wR_2 (all data)*	0.0549, 0.1127	0.1389, 0.2147

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

Supplementary material for structures has been deposited with the Cambridge Crystallographic Data Centre (CCDC nos. 1849854 (**I**) and 1849855 (**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 MIC values of the tested compounds were determined by a colorimetric method using the dye MTT [18]. A stock solution of the compound (150 μg mL^{-1}) in DMSO was prepared and graded quantities (75, 37.5, 18.8, 9.4, 4.7, 2.3, 1.2, 0.59 μg mL^{-1}) were incorporated in specified quantity of the corresponding sterilized liquid medium. A specified quantity of the medium containing the compound was poured into micro-titration plates. Suspension of the microorganism was prepared to contain approximately 1.0 × 10^5 cfu mL^{-1} and applied to microtitration plates with serially diluted compounds

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

Bond	*d*, Å	Bond	*d*, Å
		I	
Mn(1)–O(1)	1.8949(18)	Mn(1)–O(2)	1.9068(18)
Mn(1)–O(4)	1.9217(17)	Mn(1)–O(5)	2.1369(19)
Mn(1)–O(6)	2.287(2)	Mn(1)–N(1)	1.993(2)
		II	
Mn(1)–O(1)	1.888(4)	Mn(1)–O(3)	1.912(3)
Mn(1)–O(4)	2.245(4)	Mn(1)–O(6)	1.885(4)
Mn(1)–O(7)	2.189(4)	Mn(1)–N(1)	1.994(4)
Angle	ω, deg	Angle	ω, deg
		I	
O(1)Mn(1)O(2)	174.13(7)	O(1)Mn(1)O(4)	94.89(8)
O(2)Mn(1)O(4)	90.75(8)	O(1)Mn(1)N(1)	90.90(8)
O(2)Mn(1)N(1)	83.45(8)	O(4)Mn(1)N(1)	174.19(9)
O(1)Mn(1)O(5)	90.36(9)	O(2)Mn(1)O(5)	91.50(8)
O(4)Mn(1)O(5)	87.72(8)	N(1)Mn(1)O(5)	92.78(8)
O(1)Mn(1)O(6)	93.89(8)	O(2)Mn(1)O(6)	84.81(8)
O(4)Mn(1)O(6)	86.51(8)	N(1)Mn(1)O(6)	92.59(8)
O(5)Mn(1)O(6)	173.10(7)		
		II	
O(6)Mn(1)O(1)	174.35(15)	O(6)Mn(1)O(3)	93.09(16)
O(1)Mn(1)O(3)	92.53(16)	O(6)Mn(1)N(1)	83.45(16)
O(1)Mn(1)N(1)	90.91(16)	O(3)Mn(1)N(1)	175.45(16)
O(6)Mn(1)O(7)	90.51(17)	O(1)Mn(1)O(7)	89.93(17)
O(3)Mn(1)O(7)	91.24(15)	N(1)Mn(1)O(7)	91.73(15)
O(6)Mn(1)O(4)	87.98(17)	O(1)Mn(1)O(4)	91.83(17)
O(3)Mn(1)O(4)	86.33(15)	N(1)Mn(1)O(4)	90.59(15)
O(7)Mn(1)O(4)	177.06(16)		

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 mol L^{-1}, pH 7.4) containing 2 mg of MTT mL^{-1} 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 550 nm.

RESULTS AND DISCUSSION

The Schiff bases H_2L^1 and H_2L^2 were readily prepared by the condensation reaction of 2-aminophenol with 3-methoxysalicylaldehyde and 2-amino-4-chlo-

rophenol with 3-ethoxysalicylaldehyde, respectively. The manganese(III) complexes **I** and **II** were synthesized by stirring equimolar quantities of the Schiff bases with manganese salts and other materials at room temperature. Single crystals of the complexes are stable in air at ambient condition. The complexes are soluble in methanol, ethanol, acetonitrile, DMSO, DMF, but insoluble in water. The chemical formulae of the complexes have been confirmed by elemental analyses, IR spectra, and single crystal X-ray diffraction.

In the spectra of the Schiff bases and the complexes, the weak and broad bands in the range 3300–3500 cm^{-1} are assigned to the vibration of O–H bonds. The bands at 1628 and 1625 cm^{-1} characteristic of the azomethine groups presented in the free Schiff bases, were shifted to lower wave numbers at 1596 cm^{-1} for **I** and 1598 cm^{-1} for **II** and indicated involvement of the azomethine nitrogen atoms in coordination [19]. The bands at 1277–12891 cm^{-1} in the spectra of the Schiff bases are ascribed to the phenolic C–O stretching vibration. These bands are found at 1257 and 1261 cm^{-1} in the spectra of the complexes. These changes suggest that the hydroxyl groups of the Schiff bases took part in the complex formation. Further evidence of the bonding is also shown by the observation that new bands in the IR spectra of the metal complexes appear at 430–680 cm^{-1} assigned to Mn–N and Mn–O stretching vibrations.

Electronic absorption spectra of the Schiff bases and the complexes were obtained in methanol solutions. The electronic absorption spectra of the free Schiff bases displayed three bands at about 265, 345 and 450 nm, which are assigned for the $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions, respectively. In the electronic spectra of the complexes, the strong bands centered at about 250 nm are attributed to the intra-ligand $\pi \rightarrow \pi^*$ absorption peak of the Schiff base ligands. The LMCT and to some extent $\pi \rightarrow \pi^*$ bands appear at about 430 nm.

The molecular structure of complex **I** is shown in Fig. 1a. The Mn atom in the complex is in an octahedral coordination with the phenolate O and imine N atoms of the Schiff base ligand and one O atom of acac ligand defining the equatorial plane, and with the other O atom of Acac ligand and one ethanol O atom occupying the two axial positions. The Mn atom deviates from the least-squares plane defined by the four equatorial donor atoms by 0.017(3) Å. The Mn–O and Mn–N coordinate bond lengths in the complex are comparable to the corresponding values observed in other similar manganese(III) complexes with Schiff bases [20–24]. The dihedral angle between the two benzene rings of the Schiff base ligand is 8.1(5)°. In the crystal structure of the complex, adjacent two complex molecules are linked by ethanol ligands through intermolecular O–H···O hydrogen bonds (O(6)–H(6) 0.85(1), H(6)···O(2)^{i} 1.91(1) Å, O(6)···O(2)^{i} 2.749(2) Å, O(6)–H(6)···O(2)^{i} 169(4)°; symmetry code: ^{i} $1 - x$, $2 - y$, $1 - z$) to form dimers (Fig. 2a).

Table 3. Antibacterial activities of the Schiff bases and the complexes (MIC, μg mL^{-1})

Tested material	*B. subtilis*	*S. aureus*	*E. coli*	*P. fluoresence*
H_2L^1	18.8	37.5	>75	>75
H_2L^2	9.4	18.8	18.8	>75
I	4.7	9.4	37.5	>75
II	1.2	4.7	9.4	37.5
Penicillin G	2.3	4.7	>75	>75

The molecular structure of complex **II** is shown in Fig. 1b. The asymmetric unit of the compound contains a mononuclear manganese(III) complex molecule and a water molecule of crystallization. The Mn atom in the complex is in an octahedral coordination with the phenolate O and imine N atoms of the Schiff base ligand and the phenolate O atom of Esal ligand defining the equatorial plane and with the carbonyl O atoms of the Esal ligand and DMF ligand occupying the two axial positions. The Mn atom deviates from the least-squares plane defined by the four equatorial donor atoms by 0.029(3) Å. The Mn–O and Mn–N coordinate bond lengths in the complex are comparable to the corresponding values observed in other similar manganese(III) complexes with Schiff bases [20–24]. The dihedral angle between the two benzene rings of the Schiff base ligand is 7.0(6)°. In the crystal structure of the complex, the water molecules are linked to the manganese complex molecules through O–H···O hydrogen bonds (O(8)–H(8*B*) 0.85, H(8*B*)···O(1) 2.53, O(8)···O(1) 3.106(7) Å, O(8)–H(8B)···O(1) 126(5)°; O(8)–H(8*B*) 0.85, H(8*B*)···O(2) 2.50, O(8)···O(2) 2.991(9) Å, O(8)–H(8*B*)···O(2) 118(5)°; O(8)–H(8*A*) 0.85, H(8*A*)···O(3) 2.50, O(8)···O(3) 2.920(8) Å, O(8)–H(8A)···O(3) 112(5)°) (Fig. 2b).

The Schiff bases and the two manganese complexes were screened for antibacterial activities 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 (minimum inhibitory concentration, μg mL^{-1}) values of the compounds against four bacteria are listed in Table 3. Penicillin G was used as the standard drug. The Schiff base H_2L^1 shows medium activity against *B. subtilis* and *S. aureus* and no activity against *E. coli* and *P. fluorescence*. The Schiff base H_2L^2 shows strong activity against *B. subti-*

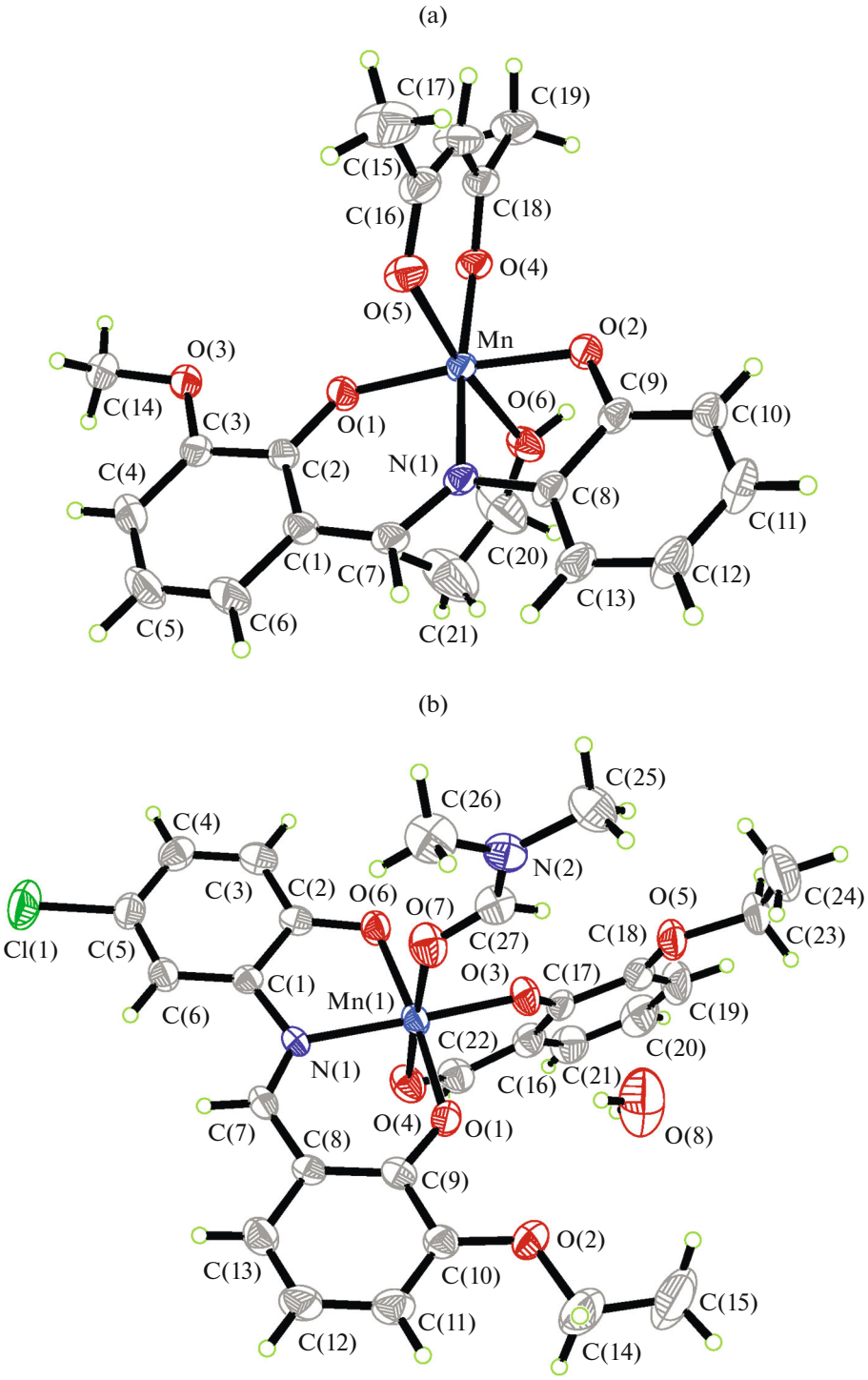


Fig. 1. A perspective view of the complex **I** (a) and **II** (b) with the atom labeling scheme. Thermal ellipsoids are drawn at the 30% probability level.

lis, medium activity against *S. aureus* and *E. coli* and no activity against *P. fluorescence*. In general, H_2L^2 has stronger activities than H_2L^1. Complex **I** has strong activity against *B. subtilis* and *S. aureus*, weak activity against *E. coli* and no activity against *P. fluorescence*. Complex **II** has strong activity against *B. subtilis*, *S. aureus,* and *E. coli* and weak activity against *P. fluorescence*. Like the Schiff bases, complex **II** has stronger

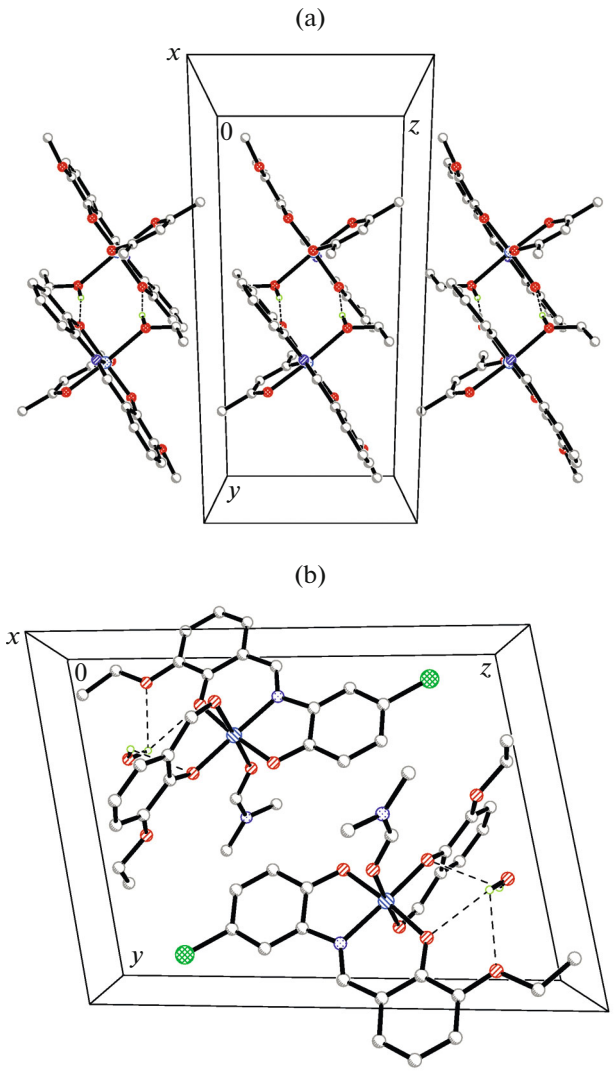


Fig. 2. Hydrogen bonds linked dimeric structure of complex **I** (a) and **II** (b), viewed along the *x* axis. Hydrogen bonds are drawn as dotted lines.

activities than complex **I**. It is noticeable that the complex **II** has the most activity against *B. subtilis* and *S. aureus* as compared to Penicillin G.

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