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Synthesis, Crystal Structures, and Biological Activity of Manganese(III) Complexes Derived from BIS-Schiff Bases[1]

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Abstract—Two new manganese(III) complexes, $[Mn^{III}L^1(Dca)(MeOH)]$ (**I**) and $[Mn^{III}L^2(N_3)_2]$ (**II**), where L^1 and L^2 are the dianionic form of *N*,*N*'-3,4-chlorophenylene-bis(5-methylsalicylaldimine) (H_2L^1) and *N*,*N*'-3,4-nitrophenylene-bis(5-methylsalicylaldimine) (H_2L^2), respectively, and Dca is dicyanoamide, have been synthesized and characterized. The complexes were characterized by elemental analyses, IR, UV-Vis spectra, molar conductivity, and single crystal X-ray diffraction (CIF files nos. 1054200 (**I**), 1054336 (**II**)). The Mn atoms in the structures are in octahedral coordination. In the crystal structure of complex **I**, molecules are linked through intermolecular O–H···N hydrogen bonds to form 1D chains running along the *x* axis. In both complexes, there exist π···π interactions among molecules. The complexes and the Schiff bases 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.

Keywords: Schiff base, manganese(III) complex, X-ray diffraction, antibacterial activity

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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 manganese(III) complexes derived from Schiff base ligands due to their important role in catalytic, magnetic and biological properties [6–10]. In addition, manganese plays an important role in metalloenzymes such as catalase [11], superoxide dismutase [12, 13], and photosystem II of green plants [14, 15]. An important aspect of Mn(III) salen type complexes is their antibacterial application [16–18]. We report here the synthesis, characterization including single crystal X-ray structures of two new manganese(III) complexes, $[Mn^{III}L^1(Dca)(MeOH)]$ (**I**) and $[Mn^{III}L^2(N_3)_2]$ (**II**), where L^1 and L^2 are the dianionic form of *N*,*N*'-3,4-chlorophenylene-bis(5-methylsalicylaldimine) (H_2L^1) and *N*,*N*'-3,4-nitrophenylene-bis(5-methylsalicylaldimine) (H_2L^2), respectively, and Dca is dicyanoamide. 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 H_2L^1 and H_2L^2 were prepared by 2 : 1 condensation of 5-methylsalicylaldehyde with 4-chloro-*o*-phenylenediamine and 4-nitro-*o*-phenylenediamine, respectively, 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 diffractometer. Electronic spectra were obtained with Lambda 35 spectrophotometer. 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 they should be handled with caution.

Synthesis of complex I. To a stirred suspension of H_2L^1 (0.379 g, 1.00 mmol) and sodium dicyanoamide (0.178 g, 2.00 mmol) in methanol (20 mL) was added

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dropwise a methanol solution (10 mL) of manganese(II) perchlorate hexahydrate (0.254 g, 1.00 mmol). After a few minutes a brown precipitate started to deposit. This was dissolved by adding the requisite amount of acetonitrile. After one hour stirring, the solution was filtered and the filtrate was kept for slow evaporation. The diffraction quality deep brown single crystals that deposited over a period of a few days were collected by filtration and washed with methanol. The yield was 291 mg (55%).

For $C_{25}H_{21}N_5O_3ClMn$

anal. calcd., %	C, 56.67	H, 3.99	N, 13.22
Found, %	C, 56.54	H, 4.10	N, 13.31

Synthesis of complex II. To a stirred suspension of H_2L^2 (0.389 g, 1.00 mmol) and sodium azide (0.130 g, 2.00 mmol) in methanol (20 mL) was added dropwise a methanol solution (10 mL) of manganese(II) perchlorate hexahydrate (0.254 g, 1.00 mmol). After a few minutes a brown precipitate started to deposit. This was dissolved by adding the requisite amount of acetonitrile. After one hour stirring, the solution was filtered and the filtrate was kept for slow evaporation. The diffraction quality deep brown single crystals that deposited over a period of a few days were collected by filtration and washed with methanol. The yield was 319 mg (60%).

For $C_{22}H_{17}N_9O_4Mn$

anal. calcd., %	C, 50.20	H, 3.26	N, 23.95
Found, %	C, 50.31	H, 3.33	N, 23.76

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 were used [20]. Structures of the complexes were solved by direct and Fourier methods and refined by full-matrix least-squares based on F^2 using SHELXL-97 package [21]. The non-hydrogen atoms were refined anisotropically. The remaining hydrogen atoms have been placed at geometrical positions with fixed thermal parameters. Crystallographic data of the complexes **I** and **II** 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. 1054200 (**I**) and 1054336 (**II**); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

Antibacterial activity. Antibacterial activity of the complexes was tested against *B. subtilis*, *S. aureus*, *S. faecalis*, *P. aeruginosa*, *E. coli*, and *E. cloacae* using MTT medium. The minimum inhibitory concentrations (MICs) of the compounds were determined by a colorimetric method using MTT dye [22]. A stock solution of the compounds (50 μg mL^{-1}) in DMSO was prepared and quantities of the compounds were incorporated in specified quantity of sterilized liquid medium. A specified quantity of the medium containing the compounds was poured into micro-titration plates. Suspension of the microorganism was prepared to contain approximately 10^5 cfu mL^{-1} and applied to micro-titration plates with serially diluted compounds in DMSO to be tested, and incubated at 37°C for 24 h for bacteria. After the MICs were visually determined on each micro-titration plate, 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 (OD) was measured with a microplate reader at 570 nm.

RESULTS AND DISCUSSION

Reaction of manganese(II) perchlorate and bis-Schiff bases in the presence of sodium dicyanoamide or sodium azide produced the mononuclear manganese(III) complexes. Clearly, aerial oxidation of manganese(II) to manganese(III) and metal assisted deprotonation of the phenolic moieties take place during the formation of the complexes. The poor conductivity of the complexes (23–35 Ω^{-1} cm^2 mol^{-1}) indicated that the ligands are coordinated to the metal center and are not dissociated in solution. The characteristic imine stretching of the complexes is observed at 1609 cm^{-1} for **I** and 1609 cm^{-1} for **II** as strong signal. In the spectra of **I**, the stretching band of methanol ligand OH group is observed at 3353 cm^{-1}. Three bands at 2290, 2235, and 2172 cm^{-1} are due to the terminal Dca ligand in the complex. In the spectra of **II**, appearance of intense band at 2026 cm^{-1} indicates the presence of azide ligand. The Schiff base ligands coordination is substantiated by the lowering of the phenolic C–O stretching band, which appears at 1179–1183 cm^{-1} in the complexes, while observed at about 1200 cm^{-1} in the free Schiff bases. Coordination of the Schiff bases is further confirmed by the appearance of weak bands in the low wave numbers 400–600 cm^{-1}, corresponding to ν(Mn–N) and ν(Mn–O). UV-Vis spectrum of the complexes exhibits two typical bands centered at 420–450 and 300–320 nm which can be assigned to phenolate → manganese(III) charge transfer and manganese(III) → imine ($d \rightarrow \pi^*$) metal to ligand charge transfer transitions, respectively. The observed magnetic moment at 300 K of the complexes

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

Parameter	Value	
	I	**II**
Molecular weight	529.86	526.39
Crystal color, habit	Brown, block	Brown, block
Crystal size, mm	0.32 × 0.30 × 0.27	0.23 × 0.22 × 0.19
Crystal system	Orthorhombic	Monoclinic
Space group	$Pna2_1$	$P2_1/c$
Unit cell dimensions:		
a, Å	14.5381(8)	12.383(2)
b, Å	10.8840(6)	7.594(1)
c, Å	14.9997(9)	24.968(2)
β, deg	90	90.00(1)
V, Å^3	2373.4(2)	2347.9(6)
Z	4	4
ρ_{calcd}, g cm^{-3}	1.483	1.489
μ, mm^{-1}	0.707	0.612
θ Range collected, deg	2.70–25.06	2.80–25.22
T_{min} and T_{max}	0.8054 and 0.8321	0.872 and 0.893
Reflections collected/unique	20190/4114	11198/3358
Observed reflections ($I \geq 2\sigma(I)$)	3593	2140
Data/restraints/parameters	4114/2/322	3358/0/327
GOOF on F^2	1.043	1.062
R_1, wR_2 ($I \geq 2\sigma(I)$)	0.0308, 0.0701	0.0896, 0.2214
R_1, wR_2 (all data)	0.0406, 0.0748	0.1404, 0.2464
Largest differences in peak/hole, e Å^{-3}	0.290/–0.174	0.475/–0.317

are 4.72 B.M., indicating that the manganese(III) centers in the complexes exist in high spin state.

Molecular structure of complex **I** is shown in Fig. 1a. The Mn centre is six coordinated by four donor atoms (N_2O_2) of the coordinated Schiff base ligand L, one oxygen atom of a methanol ligand, and one nitrogen atom of a Dca ligand. In the octahedral coordination, O(3) and N(3) are the axial ligand atoms, and O(1), O(2), N(1) and N(2) form the equatorial plane. The coordination environment of the Mn atom is slightly distorted, as evidenced from the bond lengths and angles. In the equatorial plane, the metal–ligand bond distances involving the imine nitrogens (Mn(1)–N(1) 1.990(2), Mn(1)–N(2) 1.989(3) Å) are slightly longer than the bond lengths involving phenolate oxygens (Mn(1)–O(1) 1.871(2), Mn(1)–O(2) 1.878(2) Å). As expected due to Jahn–Teller distortion of high spin manganese(III), the bond distances involving the axial Dca and methanol donor atoms (Mn(1)–N(3) 2.252(3), Mn(1)–O(3) 2.335(2) Å) significantly longer than the bond lengths involving the equatorial atoms. The *trans* angles (174.53(9)°, 170.64(8)°, and 175.28(8)°) and the *cis* angles (83.25(7)–94.02(9)°) deviate slightly from the ideal values. The coordinate bond values are in agreement with those observed in manganese(III) complexes with Schiff bases [23–25]. The average deviation (0.0562 Å) of the equatorial donor atoms and the displacement (0.0544 Å) of the metal center from the least-squares plane defined by the equatorial donor atoms indicates that the N_2O_2 cavity affords an almost perfect plane to the metal center. The Schiff base ligand is approximate planar with dihedral angel between the two methyl-substituted benzene rings of 15.9(3)°. As is commonly observed in Dca anions [26, 27], the terminal Dca ligand in the present structure adopts a ''V''-type conformation with the central C(23)N(4)C(24) angle 120.5(3)°. The carbon atoms of the Dca ligand are *sp* hybridised and almost linear, with NCN angles of 174.9(3)° and 175.2(3)°, while the C(23)N(3)Mn(1) angle, 163.5(3)°, deviates from linearity. In the crystal structure of the complex (Fig. 2a), molecules are linked through intermolecular O(3)–H(3)···N(5) hydrogen bonds (O(3)–H(3) 0.90(1),

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)	1.871(2)	Mn(1)–O(2)	1.878(2)
Mn(1)–N(1)	1.990(2)	Mn(1)–N(2)	1.989(3)
Mn(1)–N(3)	2.252(2)	Mn(1)–O(3)	2.335(2)
		II	
Mn(1)–O(1)	1.859(5)	Mn(1)–O(2)	1.873(5)
Mn(1)–N(1)	1.972(5)	Mn(1)–N(2)	2.006(5)
Mn(1)–N(3)	2.172(7)	Mn(1)–N(6)	2.521(8)
Angle	ω, deg	Angle	ω, deg
		I	
O(1)Mn(1)O(2)	93.81(9)	O(1)Mn(1)N(2)	174.53(9)
O(2)Mn(1)N(2)	91.66(9)	O(1)Mn(1)N(1)	92.85(8)
O(2)Mn(1)N(1)	170.65(8)	N(1)Mn(1)N(2)	81.69(8)
O(1)Mn(1)N(3)	90.36(10)	O(2)Mn(1)N(3)	94.02(9)
N(2)Mn(1)N(3)	89.47(10)	N(1)Mn(1)N(3)	92.49(9)
O(1)Mn(1)O(3)	87.80(10)	O(2)Mn(1)O(3)	90.44(8)
N(2)Mn(1)O(3)	91.94(9)	N(1)Mn(1)O(3)	83.25(7)
N(3)Mn(1)O(3)	175.28(8)		
		II	
O(1)Mn(1)O(2)	90.4(2)	O(1)Mn(1)N(1)	92.9(2)
O(2)Mn(1)N(1)	173.0(2)	O(1)Mn(1)N(2)	170.5(2)
O(2)Mn(1)N(2)	92.8(2)	N(1)Mn(1)N(2)	83.0(2)
O(1)Mn(1)N(3)	100.7(3)	O(2)Mn(1)N(3)	93.5(3)
N(1)Mn(1)N(3)	91.9(3)	N(2)Mn(1)N(3)	88.0(2)
O(1)Mn(1)N(6)	86.7(3)	O(2)Mn(1)N(6)	88.9(3)
N(1)Mn(1)N(6)	85.1(3)	N(2)Mn(1)N(6)	84.4(3)
N(3)Mn(1)N(6)	172.2(3)		

H(3)···N(5)^{i} 1.91(1), O(3)···N(5)^{i} 2.794(3) Å, O(3)–H(3)···N(5)^{i} 172(3)°; symmetry code: ^{i} $-1/2 + x$, $1/2 - y$, z) to form 1D chains running along the x axis.

Molecular structure of the complex **II** is shown in Fig. 1b. The structure shows that the complex having the metal center in the salen-type cavity of L^{2-}. The Mn atom is six-coordinated, with two oxygen and two nitrogen atoms of the Schiff base ligand in the equatoiral plane, and with two azido nitrogen atoms defining the axial positions. The coordination environment of the metal ion is slightly distorted octahedral. The average deviation (0.0241 Å) of the equatorial donor atoms and the displacement (0.1231 Å) of the metal center from the least-squares plane O(1)–O(2)–N(1)–N(2) indicates that the N_2O_2 cavity affords an almost perfect plane to the metal center. In the equatorial plane, the metal–ligand bond distances involving the imine nitrogens (Mn(1)–N(1) 1.972(5), Mn(1)–N(2) 2.006(5) Å) are slightly longer than the bond lengths involving phenolate oxygens (Mn(1)–O(1) 1.859(5), Mn(1)–O(2) 1.873(5) Å). As expected due to Jahn–Teller distortion of high spin manganese(III), the bond distances involving the axial azide donor atoms (Mn(1)–N(3) 2.172(7), Mn(1)–N(6) 2.521(8) Å) significantly longer than the bond lengths involving the equatorial atoms. The transoid angles (173.0(2)°, 170.5(2)°, and 172.2(3)°) and the cisoid angles (83.0(2)°–93.5(3)°) deviate slightly from the ideal values. The coordinate bond values are comparable to those in manganese(III) complexes with Schiff bases [23–25]. The Schiff base ligand is approximate planar with dihedral angel between the two methyl-substituted benzene rings of 12.9(5)°.

As shown in Fig. 2b, the complex molecules stack along the y axis via π···π interactions (Cg(1)···Cg(2)^{i} 4.706(5), Cg(3)···Cg(4)^{i} 4.694(5), Cg(3)···Cg(4)^{ii} 4.817(5), Cg(4)···Cg(4)^{ii} 4.985(5), Cg(4)···Cg(2)^{i} 3.691(5) Å, symmetry codes: ^{i} $-x$, $-y$, $-z$; ^{ii} $-x$, $1 - y$, $-z$;

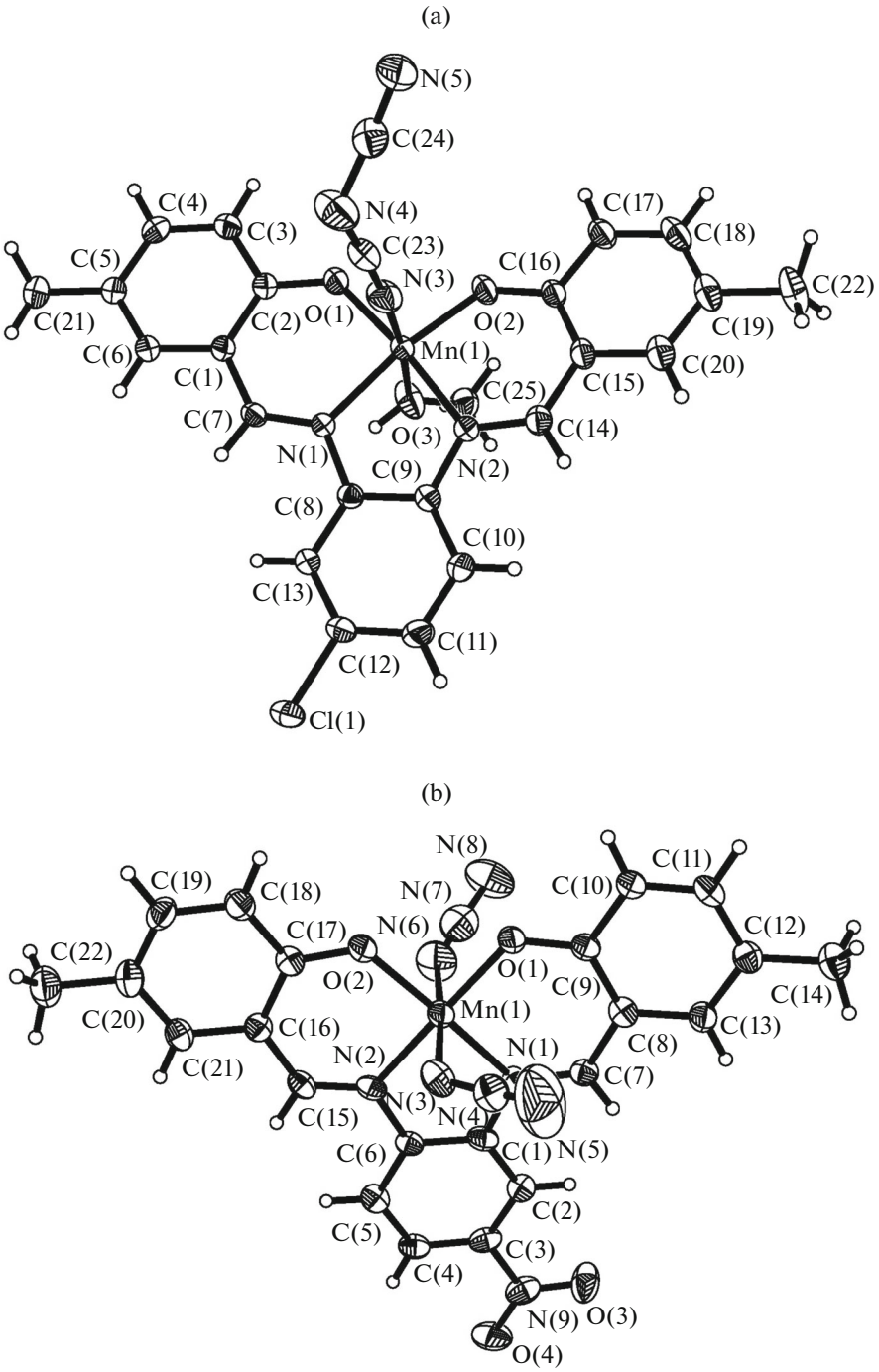

Fig. 1. Molecular structures of complexes **I** (a) and **II** (b).

Cg(1), Cg(2), Cg(3) and Cg(4) are the centroids of Mn(1)–N(1)–C(1)–C(6)–N(2), C(16)–C(17)–C(18)–C(19)–C(20)–C(21), Mn(1)–O(2)–C(17)–C(16)–C(15)–N(2), and C(1)–C(2)–C(3)–C(4)–C(5)–C(6), respectively).

The complexes and the free Schiff bases 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 compounds against the bacteria are presented in Table 3. Penicillin and Kanamycin were tested as reference drugs. Complex **I** show strong activities against all the bacteria, while complex **II** show strong activities against *B. subtilis*, *S. aureus*, *P. aeruginosa*, and *E. cloacae*, and weak activity against *St. faecalis* and medium activity against

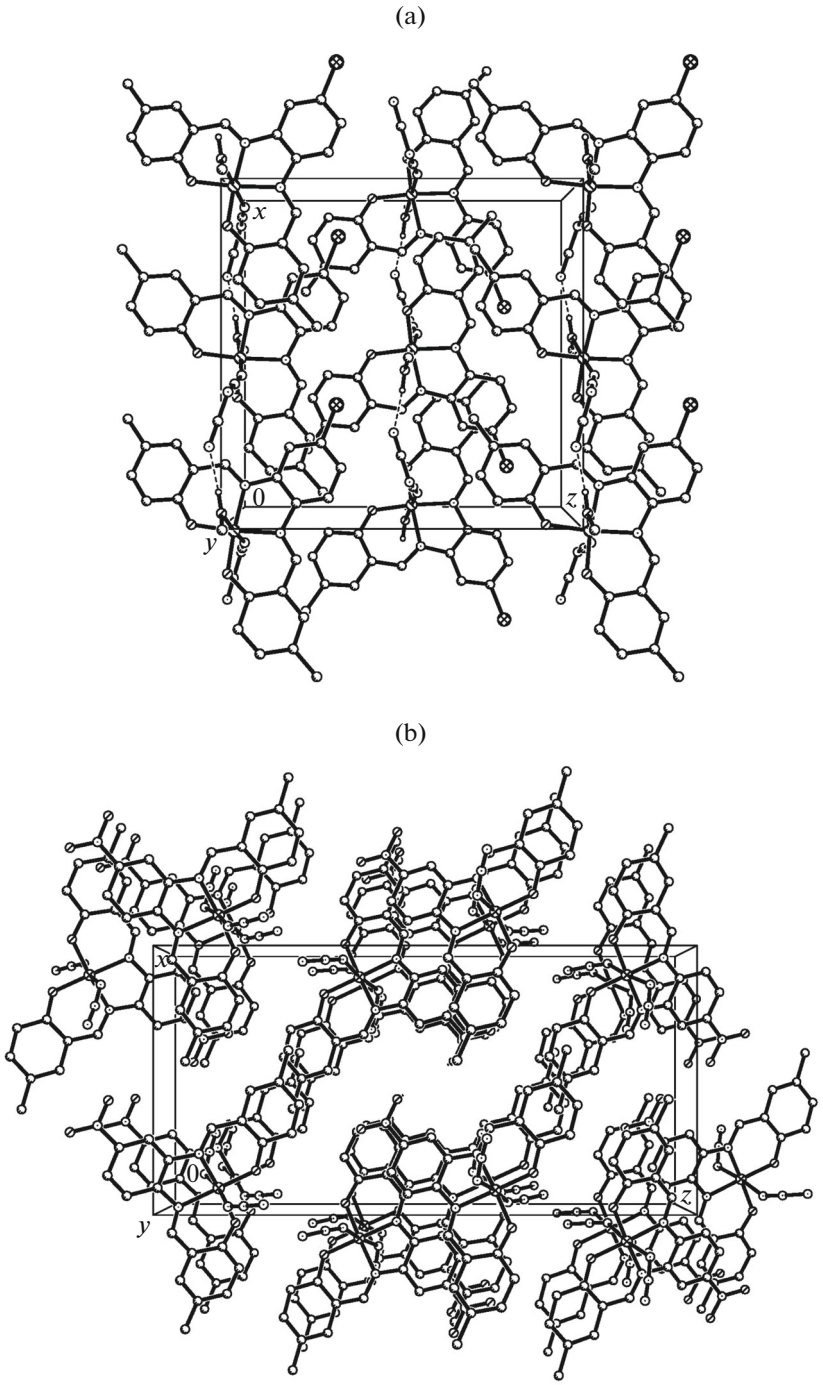

Fig. 2. Molecular packing structures of complexes **I** (a) and **II** (b). Hydrogen bonds are depicted by dashed lines.

E. coli. H_2L^1 show medium activities against *S. aureus*, *P. aeruginosa*, and strong activity against *E. cloacae*, while no activity against *B. subtilis* and *St. faecalis*. H_2L^2 show weak activities against *P. aeruginosa* and *E. cloacae*, while no activity against other bacteria strains. In general, the antibacterial activities of the two complexes are obvious higher than the free Schiff bases. Complex **I** is obvious more active than complex **II**. As a corresponding, H_2L^1 is more active than H_2L^2, which might be attributed to the contribution of the chloro-substituent group of H_2L^1.

Thus, two new manganese(III) complexes with bis-Schiff bases and pseudohalide ligands have been synthesized and characterized. Crystal structures of the complexes are described. The antibacterial assay of the free Schiff bases and the complexes shows that the

Table 3. MICs (μg mL^{-1}) of the compounds and related materials

Tested material	Gram positive			Gram negative		
	B. subtilis	*S. aureus*	*St. faecalis*	*P. aeruginosa*	*E. coli*	*E. cloacae*
I	0.39	1.56	6.25	1.56	3.12	3.12
II	1.56	6.25	25	3.12	12.5	3.12
H_2L^1	>50	12.5	>50	12.5	25	6.25
H_2L^2	>50	>50	>50	25	>50	25
Penicillin	1.56	1.56	1.56	6.25	6.25	3.12
Kanamycin	0.39	1.56	3.12	3.12	3.12	1.56

complexes, especially complex **I** are efficient drugs for most bacteria strains.

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