

Synthesis and crystal structures of manganese(III) complexes derived from bis-Schiff bases with antibacterial activity

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Abstract

Two mononuclear Mn(III) complexes, $[MnL^1(N_3)(OH_2)]$ (1) and $[MnL^2L']$ (2), where L¹ and L² are the dianionic forms of *N*,*N*'-bis(3-ethoxysalicylidene)-1,2-ethanediamine (H₂L¹) and *N*,*N*'-bis(3,5-dichlorosalicylidene)-1,4-butanediamine (H₂L²), respectively, and L' is the deprotonated form of 2,4-dichloro-6-(dimethoxymethyl)phenol (HL'), have been synthesized and characterized. The complexes were characterized by physico-chemical and spectroscopic methods. The Mn atoms in the structures are in octahedral coordination. In the crystal structure of complex (1), molecules are linked through intermolecular O–H…O hydrogen bonds to form dimers. In both complexes, there exist π … π interactions among molecules. The complexes and the Schiff bases were assayed for antibacterial activities by MTT method.

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 applications [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]. Complexes with mixed ligands usually have versatile and interesting structures [19, 20]. In order to prepare new manganese complexes and explore their biological application, we report here the synthesis and characterization including single crystal X-ray structures of two new manganese(III) complexes, $[MnL^1(N_3)(OH_2)]$ (1) and $[MnL^2L']$ (2), where L¹ and L² are the dianionic forms of *N*,*N'*-bis(3-ethoxysalicylidene)-1,2-ethanediamine (H₂L¹) and *N*,*N'*-bis(3,5-dichlorosalicylidene)-1,4-butanediamine (H₂L²), respectively, and L' is the deprotonated form of 2,4-dichloro-6-(dimethoxymethyl)phenol (HL'). 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 compounds H_2L^1 and H_2L^2 were prepared by 2:1 condensation of 3-ethoxysalicylaldehyde with ethane-1,2-diamine, and 3,5-dichlorosalicylaldehyde with butane-1,4-diamine, respectively, in methanol, according to the literature method [21]. All the other reagents and solvents were purchased from Aldrich with AR grade. FT-IR spectra were recorded as KBr pellets on a Bruker Tensor-27 spectrometer. Elemental (C, H and N) analyses were performed on a PerkinElmer 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.

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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 (1)

Table 1Crystallographic dataand refinement details forcomplexes (1) and (2)

To a stirred suspension of H_2L^1 (0.356 g, 1.00 mmol) and sodium azide (0.065 g, 1.00 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 1 h 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 272 mg (58%). Anal. Calc. (%) for C₂₀H₂₄MnN₅O₅: C, 51.2; H, 5.2; N, 14.9. Found (%): C, 51.3; H, 5.3; N, 14.7. IR data (KBr, cm⁻¹): 3378 w, 2031 s, 1606 s, 1445 m, 1372 m, 1257 w, 1185 m, 1077 s, 943 m, 862 s, 765 w, 634 w, 551 w, 439 w. UV-Vis data [methanol, λ /nm (ϵ /L mol⁻¹ cm⁻¹)]: 270 (18,355), 305

(15,270), 417 (8180). Magnetic moment: 4.72 B.M. $\Lambda_{\rm M}$ (10⁻³ M in acetonitrile): 43 Ω^{-1} cm² mol⁻¹.

Synthesis of complex (2)

To a stirred suspension of H_2L^2 (0.432 g, 1.00 mmol) and 3,5-dichlorosalicylaldehyde (0.190 g, 1.00 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 solution was formed. 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 275 mg (38%). Anal. Calc. (%) for C₂₇H₂₃Cl₆MnN₂O₅: C, 44.8; H, 3.2; N, 3.9. Found (%): C, 44.7; H, 3.2; N, 4.0. IR data (KBr, cm⁻¹): 1637 s, 1612 s, 1472 w, 1443 m, 1381 m, 1285 m, 1263 w, 1175 w, 1102 m, 1080 m, 952 m, 873 m, 771 w, 735 w, 644 w, 532 w, 478 w. UV-Vis data [methanol, λ /nm (ε /L mol⁻¹ cm⁻¹)]: 275 (16,740), 315 (12,385), 445 (7232). Magnetic moment: 4.72 B.M. Λ_{M} (10⁻³ M in acetonitrile): 20 Ω^{-1} cm² mol⁻¹.

	(1)	(2)	
Molecular weight	469.38	723.11	
Crystal color, habit	Brown, block	ck Brown, block	
Crystal size, mm	$0.18 \times 0.17 \times 0.17$	0.17 0.25×0.23×0.23	
Crystal system	Monoclinic Triclinic		
Space group	$P2_1/c$	ΡĪ	
Unit cell dimensions			
a, Á	14.2751(8)	10.3872(11)	
b, Å	22.2214(12)	11.3040(12)	
<i>c</i> , Å	13.6275(7)	13.9229(12)	
<i>a</i> , °	90	108.271(2)	
<i>β</i> , °	91.6220(10)	94.174(2)	
γ, °	90	100.946(2)	
$V, \text{\AA}^3$	4321.1(4)	1508.7(3)	
Ζ	8	2	
$ ho_{ m calcd}$, g cm ⁻³	1.443	1.592	
μ , mm ⁻¹	0.653	1.009	
θ Range collected, deg	1.43–24.58	2.72-25.50	
T_{\min} and T_{\max}	0.8916 and 0.8971	0.7864 and 0.8010	
Reflections collected/unique	21,144/7215	14,066/5582	
Observed reflections $(I \ge 2\sigma(I))$	4451	4580	
Data/restraints/parameters	7215/6/575	5582/0/372	
GOOF on F^2	0.996	1.038	
$R_1, wR_2 \ (I \ge 2\sigma(I))$	0.0413, 0.0934	0.0423, 0.0914	
R_1, wR_2 (all data)	0.0877, 0.1124	0.0559, 0.0996	
Largest differences in peak/hole, $e \text{ Å}^{-3}$	0.320/-0.315	0.354/-0.348	

X-ray structure determination

Intensity data of the complexes were collected at 298(2) K on a Bruker Apex II CCD diffractometer using graphitemonochromated Mo K_{α} radiation ($\lambda = 0.71073$ Å). For data processing and absorption correction, the packages SAINT and SADABS were used [22]. 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 [23]. The non-hydrogen atoms were refined anisotropically. The water H atoms in complex (1) were located from difference Fourier maps. The remaining hydrogen atoms have been placed at geometrical positions with fixed thermal parameters. Crystallographic data of the complexes (1) and (2) are summarized in Table 1. Selected bond lengths and angles are listed in Table 2.

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 [24]. A stock solution of the compounds (50 μ g 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 10⁵ 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: Na₂HPO₄ 12H₂O 2.9 g, KH₂PO₄ 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

Chemistry

Reactions of manganese(II) perchlorate and *bis*-Schiff bases in the presence of sodium azide or

Table 2 Selected bond distances (\AA) and angles $(^{\circ})$ for complexes (1) and (2)

(1)			
Mn(1)–O(1)	1.877(2)	Mn(1)–O(2)	1.871(2)
Mn(1)-O(3)	2.323(3)	Mn(1)–N(1)	1.978(3)
Mn(1)-N(2)	1.970(3)	Mn(1)–N(3)	2.261(3)
Mn(2)-O(6)	1.877(2)	Mn(2)–O(7)	1.881(2)
Mn(2)–O(8)	2.334(2)	Mn(2)-N(6)	1.978(2)
Mn(2)-N(7)	1.975(3)	Mn(2)-N(8)	2.269(3)
O(2)-Mn(1)-O(1)	93.45(9)	O(2)-Mn(1)-N(2)	92.20(11)
O(1)-Mn(1)-N(2)	172.67(12)	O(2)-Mn(1)-N(1)	174.71(12)
O(1)-Mn(1)-N(1)	91.66(11)	N(2)-Mn(1)-N(1)	82.59(13)
O(2)-Mn(1)-N(3)	96.02(10)	O(1)-Mn(1)-N(3)	93.04(10)
N(2)-Mn(1)-N(3)	90.99(11)	N(1)-Mn(1)-N(3)	85.04(11)
O(2)-Mn(1)-O(3)	92.64(9)	O(1)-Mn(1)-O(3)	90.75(9)
N(2)-Mn(1)-O(3)	84.33(10)	N(1)-Mn(1)-O(3)	85.94(10)
N(3)-Mn(1)-O(3)	170.31(10)	O(6)-Mn(2)-O(7)	93.86(9)
O(6)-Mn(2)-N(7)	173.90(10)	O(7)-Mn(2)-N(7)	91.89(10)
O(6)-Mn(2)-N(6)	91.42(10)	O(7)-Mn(2)-N(6)	173.13(10)
N(7)-Mn(2)-N(6)	82.69(11)	O(6)-Mn(2)-N(8)	93.37(10)
O(7)-Mn(2)-N(8)	99.09(10)	N(7)-Mn(2)-N(8)	87.72(11)
N(6)-Mn(2)-N(8)	84.96(10)	O(6)-Mn(2)-O(8)	89.51(9)
O(7)-Mn(2)-O(8)	92.72(9)	N(7)-Mn(2)-O(8)	88.18(10)
N(6)-Mn(2)-O(8)	82.93(9)	N(8)-Mn(2)-O(8)	167.61(10)
(2)			
Mn(1)-O(1)	1.8789(18	S) Mn(1)–O(2)	1.9308(19)
Mn(1)-O(3)	1.8860(19) Mn(1)–O(4)	2.3824(18)
Mn(1)-N(1)	2.178(2)	Mn(1)-N(2)	2.037(2)
O(1)-Mn(1)-O(3)	91.03(9)	O(1)-Mn(1)-O(2)	91.75(9)
O(3)-Mn(1)-O(2)	169.77(8)	O(1)-Mn(1)-N(2)	174.46(9)
O(3)-Mn(1)-N(2)	87.89(9)	O(2)-Mn(1)-N(2)	88.40(9)
O(1)-Mn(1)-N(1)	90.24(8)	O(3)-Mn(1)-N(1)	98.60(8)
O(2)-Mn(1)-N(1)	91.23(8)	N(2)-Mn(1)-N(1)	95.29(9)
O(1)-Mn(1)-O(4)	87.98(7)	O(3)-Mn(1)-O(4)	84.63(7)
O(2)-Mn(1)-O(4)	85.63(7)	N(2)-Mn(1)-O(4)	86.51(8)
N(1)-Mn(1)-O(4)	176.34(8)		

3,5-dichlorosalicylaldehyde produced the mononuclear manganese(III) complexes (Scheme 1). Clearly, aerial oxidation of manganese(II) to manganese(III) and metal-assisted deprotonation of the phenolic moieties takes place during the formation of the complexes. The low solution conductivities of the complexes (20–43 Ω^{-1} cm² mol⁻¹) indicated that the ligands are coordinated to the metal center and are not dissociated in solution.

Spectroscopy

The characteristic imine stretching of the complexes is observed at $1606-1612 \text{ cm}^{-1}$ as a strong signal. In the spectra of (1), the stretching band of water ligand OH groups is observed at 3378 cm⁻¹, and the characteristic band of



Scheme 1 Synthetic procedures for the complexes

the azide ligand is observed at 2031 cm^{-1} . In the spectra of (2), the strong band at 1637 cm^{-1} indicates the presence of the carbonyl group. The Schiff base ligands coordination is substantiated by the lowering of the phenolic C-O stretching band, which appears at 1180-1185 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 410-445 and 305-315 nm which can be assigned to phenolate \rightarrow manganese(III) charge transfer and manganese(III) \rightarrow imine $(d \rightarrow \pi^*)$ metal to ligand charge transfer transitions, respectively. The observed magnetic moment at 300 K of the complexes is 4.72 B.M., indicating that the manganese(III) centers in the complexes exist in high spin state.

Structure description of complex (1)

The molecular structure of complex (1) is shown in Fig. 1. The asymmetric unit of the complex contains two $[MnL^1(N_3)(OH_2)]$ molecules. The Mn center is six coordinated by four donor atoms (N_2O_2) of the coordinated Schiff base ligand, one oxygen atom of a water ligand and one nitrogen atom of an azide ligand. In the octahedral coordination, O(3) and N(3), as well as O(8) and N(8) are the

axial ligand atoms, and O(1), O(2), N(1) and N(2), as well as O(6), O(7), N(6) and N(7) form the equatorial plane. The coordination environment of the Mn atoms is slightly distorted, as evidenced from the bond lengths and angles. In the equatorial plane, the metal-ligand bond distances involving the imine nitrogens are slightly longer than the bond lengths involving phenolate oxygens. As expected due to Jahn-Teller distortion of high spin manganese(III), the bond distances involving the axial donor atoms are significantly longer than the bond lengths involving the equatorial atoms. The *trans* angles $(167.61(10)^{\circ}-174.71(12)^{\circ})$ and the cis angles (82.59(13)°-99.09(10)°) deviate slightly from the ideal values. The coordinate bond values are in agreement with those observed in other manganese(III) complexes with Schiff bases [25-28]. The average deviation (0.029(5))Å for Mn(1) and 0.021(5) Å for Mn(2)) of the equatorial donor atoms and the displacement (0.048(3) Å for Mn(1))and 0.051(3) Å for Mn(2)) of the metal center from the leastsquares plane defined by the equatorial donor atoms indicates that the N₂O₂ cavity affords an almost perfect plane to the metal center. The Schiff base ligands are approximate planar with dihedral angels between the benzene rings of 1.1(3)° and 2.3(3)°.

Fig. 1 Molecular structure of complex (1)





Fig. 2 Molecular structure of complex (2)

Structure description of complex (2)

Molecular structure of complex (2) is shown in Fig. 2. The Mn center is six coordinated by four donor atoms (N_2O_2) of the coordinated Schiff base ligand, one phenolate oxygen atom and one ether oxygen atom of the ligand 2,4-dichloro-6-(dimethoxymethyl)phenolate. In the octahedral coordination,

N(1) and O(4) are the axial ligand atoms, and O(1), O(2), N(2) and O(3) form the equatorial plane. The coordination environment of the Mn atom is slightly distorted, as evidenced from the bond lengths and angles. As expected due to Jahn-Teller distortion of high spin manganese(III), the bond distances involving the axial donor atoms are significantly longer than the bond lengths involving the equatorial atoms. The trans angles $(169.77(8)^{\circ}-176.34(8)^{\circ})$ and the cis angles $(84.63(7)^{\circ}-98.60(8)^{\circ})$ deviate slightly from the ideal values. The coordinate bond values are in agreement with those observed in manganese(III) complexes with Schiff bases [25–28]. The average deviation (0.034(3) Å) of the equatorial donor atoms and the displacement (0.128(2))A) 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 dihedral angel between the two benzene rings of the Schiff base ligand is 74.2(5)°.

Antibacterial activity of the compounds

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

Table 3	MICs ($\mu g m L^{-1}$) of
the com	pounds and related
material	s

Tested material	Gram positive			Gram negative		
	B. subtilis	S. aureus	St. faecalis	P. aeruginosa	E. coli	E. cloacae
(1)	12.5	6.25	>50	25	> 50	> 50
(2)	1.56	0.78	12.5	3.12	6.25	25
H_2L^1	>50	25	> 50	> 50	> 50	> 50
H_2L^2	25	6.25	> 50	25	12.5	> 50
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

E. cloacae) by MTT methods. The MICs of the compounds against the bacteria are presented in Table 3. Penicillin and Kanamycin were tested as reference drugs. Complex (1) shows strong activity against S. aureus, medium activities against B. subtilis and P. aeruginosa and no activity against St. faecalis, E. coli and E. cloacae. Complex (2) shows strong activities against B. subtilis, S. aureus, P. aeruginosa and E. coli, medium activities against St. faecalis and E. *cloacae*. H_2L^1 shows medium activity against S. *aureus*, while no activity against the remaining bacteria strains. H₂L² shows strong activity against S. aureus, medium and weak activities against B. subtilis, P. aeruginosa and E. coli, while no activity against the remaining bacteria strains. In general, the antibacterial activities of the two complexes are obviously higher than the free Schiff bases. Complex (2) is more active than complex (1). Similarly, H_2L^2 is more active than H_2L^1 , which might be attributed to the contribution of the chloro-substituent groups of H_2L^2 .

Conclusion

In summary, two new manganese(III) complexes with *bis*-Schiff bases have been synthesized and characterized. Crystal structures of the complexes are described. Antibacterial assays of the free Schiff bases and the complexes show that the complexes, especially complex **2**, are effective against most of the tested bacteria strains.

Supplementary materials

The crystal data for the complexes have been deposited with the Cambridge CrystallographicData Centre (CCDC nos. 1864189 (1) and 1864190 (2); deposit@ccdc.cam.ac.uk orhttp://www.ccdc.cam.ac.uk).

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