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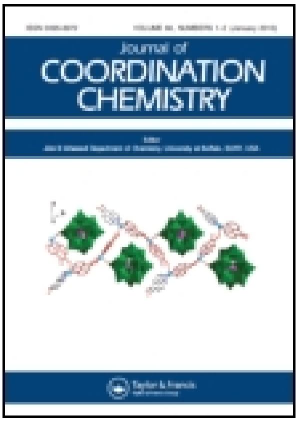

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Synthesis and crystal structures of N,N'-bis(5-fluoro-2-hydroxybenzylidene)ethane-1,2-diamine and its dinuclear manganese(III) complex with antibacterial activities

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Synthesis and crystal structures of *N,N′*-bis(5-fluoro-2-hydroxybenzylidene)ethane-1,2-diamine and its dinuclear manganese(III) complex with antibacterial activities

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A new dinuclear manganese complex, $[Mn_2L_2(N_3)_2]$, was prepared by reaction of bis-Schiff base *N,N′*-bis(5-fluoro-2-hydroxybenzylidene)ethane-1,2-diamine (H_2L) with manganese acetate and sodium azide in methanol. Both the Schiff base and the complex were characterized by physico-chemical methods and single crystal X-ray determination. The Schiff base crystallized in the orthorhombic space group *Pbca* with unit cell dimensions $a = 7.3191(7)$ Å, $b = 6.0948(6)$ Å, $c = 35.382(3)$ Å, $V = 1578.3(3)$ Å^3, $Z = 4$, $R_1 = 0.0481$, $wR_2 = 0.1488$. The manganese complex crystallized in the monoclinic space group $P2_1/c$ with unit cell dimensions $a = 8.802(1)$ Å, $b = 14.928(2)$ Å, $c = 14.478(2)$ Å, $\beta = 105.517(2)°$, $V = 1833.0(4)$ Å^3, $Z = 2$, $R_1 = 0.0768$, $wR_2 = 0.1640$. There are crystallographic inversion centers in both the ligand and the complex. The ligand coordinates to Mn through all the phenolate oxygens and imino nitrogens. The two Mn in the complex are bridged by two phenolate oxygens with separation of 3.549(1) Å. Each Mn is octahedral with four donors of the Schiff base ligand defining the equatorial plane, and with one azido nitrogen and one phenolate oxygen of an adjacent Schiff base ligand occupying the axial positions. The Schiff base and the complex were tested *in vitro* for their antibacterial activities.

Keywords: Schiff base; Manganese; Synthesis; Crystal structure; Antibacterial activity

1. Introduction

Schiff bases are readily synthesized by condensation reaction of carbonyl compounds with primary amines [1, 2]. Schiff bases have been widely investigated for biological activities, such as antibacterial and antitumor activities [3, 4], biomimetic catalytic properties [5], etc. Metal complexes of Schiff bases have also received attention. These complexes play an important role in the development of coordination chemistry related to catalysis and enzymatic reactions, magnetism and molecular architectures [6–8], and also exhibit interesting biological activities [9–11].

A number of Schiff bases derived from salicylaldehyde and ethane-1,2-diamine and their complexes have been reported [12–14]. Most show versatile biological properties, especially antibacterial activities [15, 16]. It was reported that compounds bearing one or more halogens on the aromatic ring have improved antibacterial and antifungal activities [17].

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Azide is a versatile ligand for preparation of complexes with terminal or bridging coordination [18–20]. In the present work, we choose a fluoro-containing bis-Schiff base, *N,N'*-bis(5-fluoro-2-hydroxybenzylidene)ethane-1,2-diamine (H_2L), as ligand to prepare $[Mn_2L_2(N_3)_2]$. Only five complexes derived from H_2L have been reported [21–23]. The antibacterial activities against *Bacillus subtilis*, *Staphylococcus aureus*, *Escherichia coli*, and *Pseudomonas fluorescens* were evaluated for the Schiff base and the manganese complex (scheme 1).

2. Experimental

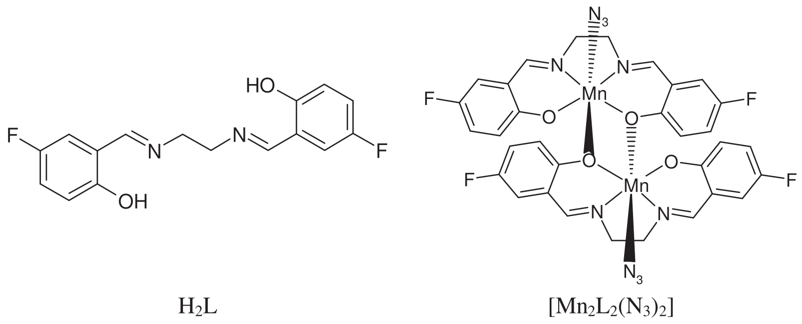
2.1. *Materials and measurements*

5-Fluoro-2-hydroxybenzaldehyde, ethane-1,2-diamine, and sodium azide of AR grade were obtained from Aldrich and used as received. Elemental analyzes were performed using a Perkin–Elmer 240C analytical instrument. Infrared spectra were recorded on a Nicolet 5DX FT-IR spectrophotometer with KBr pellets. ^{1}H NMR spectra were recorded on a Bruker instrument at 400 MHz. UV–vis spectra were recorded on a JASCO V-570 spectrophotometer. Molar conductance was measured with a Shanghai DDS-11A conductometer.

Caution! Although no problems were encountered in our work, compounds containing azide are potentially explosive. Therefore, only a small amount of the materials should be used and they should be handled with care.

2.2. *Synthesis of H_2L*

H_2L was prepared by refluxing 5-fluoro-2-hydroxybenzaldehyde (2.80 g, 0.02 M) and ethane-1,2-diamine (0.60 g, 0.01 M) in 30 mL methanol for half an hour. The solution was evaporated to give yellow powder, which was washed three times with methanol and dried in air. Yield: 93%. Anal. Calcd for $C_{16}H_{14}F_2N_2O_2$ (%): C, 63.2; H, 4.6; N, 9.2. Found: C, 63.0; H, 4.7; N, 9.3. ^{1}H NMR (d^6-DMSO): δ: 2.32 (s, 6H), 3.93 (m, 4H), 6.87 (d, 2H), 7.13 (d, 2H), 7.51 (s, 2H), 8.57 (s, 2H), 11.83 (s, 2H). ^{13}C NMR (d^6-DMSO): δ: 21.2, 61.5, 116.8, 124.5, 130.9, 131.4, 132.5, 158.1, 158.5. Single crystals of the compound, suitable for X-ray diffraction, were obtained by slow evaporation of methanolic solution of the compound in air.



Scheme 1. The Schiff base ligand and the complex.

2.3. *Synthesis of [$Mn_2L_2(N_3)_2$]*

To a methanolic solution (10 mL) of H_2L (91.3 mg, 0.3 mM) and sodium azide (19.5 mg, 0.3 mM) in methanol (20 mL), solid manganese acetate (73.5 mg, 0.3 mM) was added with stirring. The mixture was stirred for half an hour and filtered. The filtrate was kept undisturbed at room temperature to slowly evaporate for a few days, generating deep brown crystals suitable for X-ray diffraction. Crystals were isolated by filtration and dried in air. Yield 45% with respect to H_2L. Anal. Calcd for $C_{36}H_{36}Mn_2N_{10}O_4$ (%): C, 55.2; H, 4.6; N, 17.9. Found: C, 55.3; H, 4.7; N, 17.8.

2.4. *X-ray crystallography*

Suitable single crystals with high quality of the Schiff base and the complex were selected and mounted on a Bruker Smart 1000 CCD area-detector diffractometer with graphite monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å). Diffraction data for the compounds were collected by ω scan at 298(2) K. Data reduction and cell refinement were performed by SMART and SAINT [24]. Empirical absorption correction was applied by using SADABS [25]. The structures were solved by direct methods and refined with full-matrix least-squares using the SHELXL97 package [26]. The non-H atoms in the structures were subjected to anisotropic refinement. Hydrogens were located geometrically and treated with riding mode. Crystallographic data and experimental details for the complexes are summarized in table 1. Selected bond lengths and angles for the complexes are listed in table 2.

2.5. *Antibacterial test*

Antibacterial activities of the Schiff base and the complex were tested *in vitro* against *B. subtilis*, *S. aureus*, *E. coli*, and *P. fluorescens* using MH medium (Mueller–Hinton medium: casein hydrolysate 17.5 g, soluble starch 1.5 g, beef extract 1000 mL). The minimum inhibitory concentrations (MIC) of the test compounds were determined by a colorimetric method using the dye MTT (3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide) [27]. A solution of the compound (50 $\mu g\, mL^{-1}$) in DMSO was prepared and graded quantities of the test compounds were incorporated in specified quantity of sterilized liquid MH medium. A specified quantity of the medium containing the compound was poured into microtitration plates. Suspension of the microorganism was prepared to contain about 10^5 colony forming units cfu mL^{-1} and applied to microtitration plates with serially diluted compounds in DMSO to be tested and incubated at 37 °C for 24 h. After MICs were visually determined on each of the microtitration plates, 50 µL of PBS (Phosphate Buffered Saline 0.01 M 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 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 isopropyl alcohol containing 5% 1.0 M 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. The observed MICs are presented in table 3.

3. Results and discussion

The manganese(III) complex was prepared by reaction of equimolar quantities of the Schiff base ligand, manganese acetate, and sodium azide in methanol. As usually observed for

Table 1. Crystallographic and experimental data for H_2L and $[Mn_2L_2(N_3)_2]$.

Complex	H_2L	$[Mn_2L_2(N_3)_2]$
Chemical formula	$C_{18}H_{20}N_2O_2$	$C_{36}H_{36}Mn_2N_{10}O_4$
Formula weight	296.4	782.6
T (K)	298(2)	298(2)
λ(Mo-Kα) (Å)	0.71073	0.71073
Color; shape	Yellow; block	Blue; block
Crystal size (mm^3)	0.27 × 0.23 × 0.23	0.15 × 0.13 × 0.10
Crystal system	Orthorhombic	Monoclinic
Space group	*Pbca*	$P2_1/c$
a (Å)	7.3191(7)	8.802(1)
b (Å)	6.0948(6)	14.928(2)
c (Å)	35.382(3)	14.478(2)
β (°)	90	105.517(2)
V (Å^3)	1578.3(3)	1833.0(4)
Z	4	2
ρ (g/cm^3)	1.247	1.418
μ (Mo-Kα) (mm^{-1})	0.082	0.742
θ Range (°)	1.15–25.50	2.00–25.49
F(0 0 0)	632	808
T_{min} and T_{max}	0.9782 and 0.9814	0.8968 and 0.9295
No. of measured reflections	7437	9103
No. of unique reflections	1472	3335
No. of observed reflections	1084	1623
Parameters/restraints	102/0	237/0
R_{int}	0.0265	0.0821
Goodness of fit on F^2	1.107	0.970
R_1, wR_2 [$I \geq 2\sigma(I)$][a]	0.0481, 0.1488	0.0768, 0.1640
R_1, wR_2 (all data)[a]	0.0733, 0.1779	0.1673, 0.2053

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

Table 2. Selected bond lengths (Å) and angles (°) for H_2L and $[Mn_2L_2(N_3)_2]$.

H_2L			
Bond lengths			
C8–N1	1.274(3)	C9–N1	1.452(3)
Bond angles			
C1–C8–N1	122.7(2)	C8–N1–C9	118.5(2)
$[Mn_2L_2(N_3)_2]$			
Bond lengths			
C8–N1	1.258(7)	C9–N1	1.498(8)
C11–N2	1.276(7)	C10–N2	1.476(9)
Mn1–O1	1.859(4)	Mn1–O2	1.900(4)
Mn1–N1	1.978(5)	Mn1–N2	1.979(5)
Mn1–N3	2.136(7)		
Bond angles			
C1–C8–N1	126.6(6)	C8–N1–C9	122.1(6)
C12–C11–N2	123.5(6)	C10–N2–C11	123.0(6)
O1–Mn1–O2	94.12(17)	O1–Mn1–N1	92.22(19)
O2–Mn1–N1	161.2(2)	O1–Mn1–N2	170.4(2)
O2–Mn1–N2	88.6(2)	N1–Mn1–N2	82.5(2)
O1–Mn1–N3	97.8(2)	O2–Mn1–N3	97.3(2)
N1–Mn1–N3	99.3(2)	N2–Mn1–N3	90.9(2)

manganese complexes, Mn^{II} underwent aerial oxidation to Mn^{III} in the synthetic route. The molar conductivity of the complex measured in methanol at concentration of 10^{-3} M is 21 Ω^{-1} cm^2 M^{-1}, indicating the non-electrolytic nature of the complex in solution [28].

Table 3. Antibacterial activities.

	MIC (μg mL^{-1})			
	B. subtilis	*E. coli*	*P. fluorescens*	*S. aureus*
H_2L	50	25	50	25
$[Mn_2L_2(N_3)_2]$	12.5	3.12	12.5	6.25
Penicillin	1.3	>100	>100	2.1

3.1. *Crystal structure description*

Figures 1 and 2 give perspective views of H_2L and the manganese complex, respectively. The Schiff base possesses crystallographic inversion center symmetry, with the inversion center located at the midpoint of C9 and C9A. There are two intramolecular O–H···N hydrogen bonds in the free Schiff base, which are absent in the complex. The dihedral angles between the two benzene rings are 0° for H_2L and 9.0(3)° for the complex. The torsion angle of N1–C9–C9A–C8A in H_2L is 0°, yet, in the complex, the corresponding torsion angle of N1–C9–C10–N2 is –47.9(3)°. The bond values of H_2L are within normal ranges when compared to other similar bis-Schiff bases [29, 30].

The complex is a centrosymmetric dinuclear manganese(III) compound, with the inversion center located at the midpoint of the two Mn ions. The Mn are bridged by two phenolate oxygens, with separation of 3.549(1) Å. The Schiff base ligand forms one five- and two six-membered chelate rings with bite angles of 82.5(2)°, 92.2(2)°, and 88.6(2)°. Each Mn is octahedral with the four donors of the Schiff base defining the equatorial plane, and with one azido nitrogen and one phenolate oxygen of an adjacent Schiff base occupying the axial positions. The displacement of Mn from the equatorial plane towards the axial azide ligand is 0.217(2) Å. The axial Mn–O and Mn–N bonds are much longer than those in the equatorial plane, caused by Jahn–Teller distortion. The *cis* bond angles are 80.6(2)–99.3(2)° and the *trans* bond angles are 161.2(2)–170.4(2)°. The Mn–O and Mn–N bond lengths in the complex are comparable to the values observed in Schiff base manganese(III) complexes [31, 32].

Coordination of the Schiff base to Mn is shown by the change of related bond lengths and angles. The bond length of C8–N1 [1.258(7) Å] is much shorter than that of 1.274(3) Å in H_2L, but the bond length of C11–N2 [1.276(7) Å] is equal to that of H_2L. The bond lengths of C2–O1 [1.338(6) Å] and C13–O2 [1.335(6) Å] in the complex are slightly shorter than that of C2–O1 [1.345(2) Å] in H_2L.

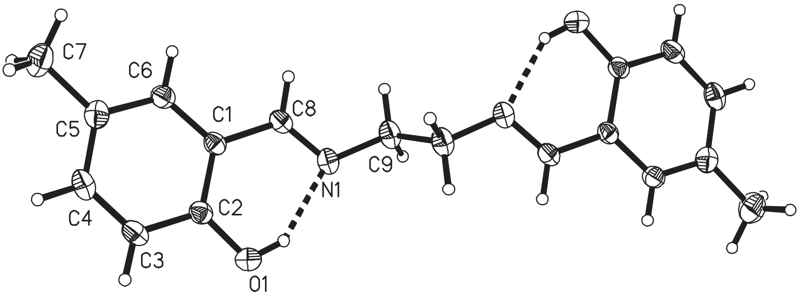


Figure 1. Molecular structure of H_2L. Displacement is drawn at the 30% probability level. Unlabeled atoms are at the symmetry position $1 - x$, $1 - y$, $1 - z$.

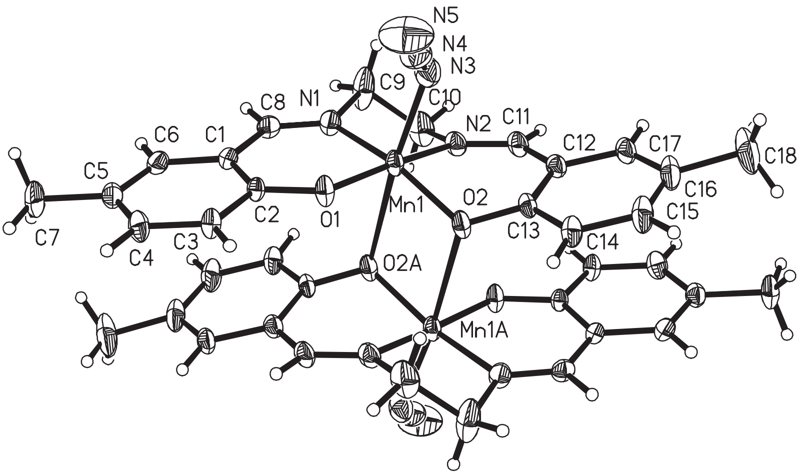


Figure 2. Molecular structure of $[Mn_2L_2(N_3)_2]$. Displacement is drawn at the 30% probability level. Atoms labeled with the suffix A or unlabeled are at the symmetry position $1 - x$, $2 - y$, $1 - z$.

3.2. *IR and electronic spectra*

IR spectra of H_2L and the manganese complex also provide information about the metal-ligand bonding. The weak and broad absorption centered at 3450 cm^{-1} is assigned to stretch of the phenolic O–H of the free Schiff base, absent in the spectrum of the complex, indicating coordination through deprotonated phenolate oxygen. Several other bands from 2900 to 3150 cm^{-1} are assigned to absorption of CH. The phenolic ν(C–O) in the spectrum of the Schiff base is a medium band at 1277 cm^{-1}. However, in the spectrum of the complex, the absorption appears at 1290 cm^{-1}. The strong band at 1637 cm^{-1} of the free Schiff base is assigned to the azomethine group, $\nu_{C=N}$ [33], which is observed at 1623 cm^{-1} for the complex, indicating coordination of the imino nitrogen. The spectrum of the complex exhibits a very strong and sharp absorption at 2034 cm^{-1}, corresponding to the asymmetric stretch of terminal azide. The newly observed bands in the region 450–620 cm^{-1} can be assigned to the Mn–O and Mn–N bonds.

The electronic spectra of the compounds in acetonitrile were recorded from 200 to 800 nm. The intense absorption bands at 232 and 278 nm may be assigned to intra-ligand π–π transitions. The weak absorptions centered at 312 and 413 nm may be assigned to the phenolate of Schiff base ligand to Mn center charge transfer band (LMCT).

3.3. *Antibacterial activities*

The Schiff base H_2L and the complex were screened *in vitro* for antibacterial activities against *B. subtilis*, *S. aureus*, *E. coli*, and *P. fluorescens* by the MTT method. The MICs of the compounds against the bacteria are presented in table 3. Penicillin was used as a reference.

The Schiff base H_2L shows moderate antibacterial activities against all four bacteria; the complex has stronger activities against the bacteria than the free Schiff base. Even though the complex has from moderate to strong activities against *B. subtilis* and *S. aureus*, it is much weaker than Penicillin. But, for *E. coli* and *P. fluorescens* the complex is superior to

Penicillin. The trends in the present work accord with those in the literatures that metal complexes usually have stronger antibacterial activities than their corresponding Schiff base ligands [34–38].

4. Conclusions

A new dinuclear manganese complex has been prepared and characterized. Coordination of the bis-Schiff base *N,N′*-bis(5-fluoro-2-hydroxybenzylidene)ethane-1,2-diamine to Mn was investigated by infrared spectra and single crystal X-ray determination. The Schiff base coordinates to Mn through phenolate oxygen and imino nitrogen with azide coordinating to Mn terminal. The antibacterial activities of the Schiff base and the complex were assayed. The results indicate that the complex is a potential antibacterial material.

Supplementary material

CCDC reference numbers 942635 for H_2L and 942636 for the manganese complex contain the supplementary crystallographic data for this paper. These data can be obtained free of charge at www.ccdc.cam.ac.uk, or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44 1223 336 033; E-mail: deposit@ccdc.cam.ac.uk.

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