# Synthesis, Structures, and Antibacterial Activities of Two Iron(III) Complexes with Schiff Bases<sup>1</sup>

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**Abstract**—Two Schiff base iron(III) complexes,  $[FeL^{1}(AHA)] \cdot H_{2}O(I)$  and  $[Fe(L^{2})_{2}] \cdot ClO_{4}(II)$ , where AHA is the deprotonated form of acetohydroxamic acid, and L<sup>1</sup> and L<sup>2</sup> are the anionic form of *N*,*N*'-bis(3-ethoxysalicylidene)propane-1,2-diamine and 2-[1-(2-aminopropylimino)ethyl]phenol, respectively, have been synthesized and characterized by physical chemical methods and single crystal X-ray diffraction. Crystallographic data for I: orthorhombic, space group *Iba2*, *a* = 20.781(3), *b* = 23.527(3), *c* = 10.071(2) Å<sub>3</sub>, *V* = 4923.7(12) Å<sup>3</sup>, *Z* = 8, *R*<sub>1</sub> = 0.0374, *wR*<sub>2</sub> = 0.0900. Crystallographic data for II: triclinic, space group *P* i, *a* = 12.748(1), *b* = 13.401(1), *c* = 19.007(1) Å,  $\alpha$  = 106.623(2)°,  $\beta$  = 97.462(2)°,  $\gamma$  = 112.543(2)°, *V* = 2784.9(4) Å<sup>3</sup>, *Z* = 4, *R*<sub>1</sub> = 0.0892, *wR*<sub>2</sub> = 0.2434. X-ray crystal structural study indicated that the coordination environment around each Fe atom in the complexes is a six-coordinated distorted octahedron. The antibacterial activities of the complexes were assayed.

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### **INTRODUCTION**

Iron is an important element for biological processes of human beings [1-3]. Iron is also an essential cofactor in metalloenzymes [4]. Its importance in DNA synthesis, control of gene expression, and induction of cell apoptosis is becoming better understood [5]. Schiff bases derived from benzaldehydes with various organic amines are important ligands in the coordination of transition metal atoms. Iron complexes with Schiff bases have been received particular interest for their biological effects, especially antibacterial and antitumor activities [6-10]. However, when compared to other transition metal ions, the number of complexes bearing iron centers is much less. In the present work, two new iron(III) complexes,  $[FeL^{1}(AHA)] \cdot H_{2}O(I)$ and  $[Fe(L^2)_2] \cdot ClO_4$  (II), where AHA is the deprotonated form of acetohydroxamic acid, and  $L^1$  and  $L^2$ are the anionic form of N,N-bis(3-ethoxysalicylidene)propane-1,2-diamine ( $H_2L^1$ ) and 2-[1-(2aminopropylimino)ethyllphenol (HL<sup>2</sup>), respectively, have been synthesized, characterized, and investigated for their antibacterial activities.

# **EXPERIMENTAL**

All chemical reagents and solvents were of analytical grade and were obtained from Sigma-Aldrich. Methanol was dried over molecular sieves (4 N) prior to use. Elemental analyses were performed on a PerkinElmer 2400 II elemental analyzer. Infrared spectra were recorded on a PerkinElmer RX I FT-IR spectrophotometer with KBr discs. TG–DTA analysis was carried out under nitrogen with a heating rate of 10°C min<sup>-1</sup> using a Perkin Elmer Pyris Diamond TG/DTA analyzer.

Iron perchlorate is potentially explosive, only small quantity should be used and handled with great care.

Synthesis of the Schiff bases. The Schiff bases  $H_2L^1$ and  $HL^2$  were synthesized by refluxing hot ethanolic solution (30 mL) of propane-1,2-diamine (0.01 mol) with 3-ethoxysalicylaldehyde (0.02 mol) and 2-acetylphenol (0.02 mol), respectively for about 1 h. The precipitates formed during reflux were filtered, washed with cold EtOH, and recrystallized from hot EtOH. The yields were 83 and 77% for  $H_2L^1$  and  $HL^2$ , respectively.

For  $C_{21}H_{26}N_2O_4(H_2L^1)$ anal. calcd., %: C, 68.1; H, 7.1; N, 7.6. Found, %: C, 67.9; H, 7.2; N, 7.5. For C<sub>11</sub>H<sub>16</sub>N<sub>2</sub>O (HL<sup>2</sup>) anal. calcd., %: C, 68.7; N, 14.6. H, 8.4; Found, %: C, 68.6; H, 8.3; N, 14.4.

Synthesis of complex I. A methanolic solution (10 mL) of  $H_2L^1$  (0.1 mmol, 37.0 mg) was mixed with a methanolic solution (10 mL) of Fe(ClO<sub>4</sub>)<sub>3</sub> (0.1 mmol, 37.2 mg) and acetohydroxamic acid (0.1 mmol, 7.5 mg), and refluxed in a water bath for

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1 h. Single crystals suitable for X-ray diffraction were obtained by slow evaporation of the solution in air for a few days. The yield was 43%.

For $C_{23}H_{30}N_3O_7Fe$					
anal. calcd., %:	C, 53.5;	Н, 5.9;	N, 8.1.		
Found, %:	C, 53.7;	Н, 6.0;	N, 8.1.		

Synthesis of complex II. A methanolic solution (10 mL) of HL<sup>2</sup> (0.1 mmol, 19.2 mg) was mixed with a methanolic solution (10 mL) of Fe(ClO<sub>4</sub>)<sub>3</sub> (0.1 mmol, 37.2 mg), and refluxed in a water bath for 1 h. Single crystals suitable for X-ray diffraction were obtained by slow evaporation of the solution in air for a few days. The yield was 65%.

For $C_{22}H_{30}N_4O_6ClFe$					
anal. calcd., %:	C, 49.1;	Н, 5.6;	N, 10.4.		
Found, %:	C, 49.0;	Н, 5.7;	N, 10.2.		

Single crystal X-ray diffraction. X-ray data for the complexes were collected on a Bruker SMART APEXII diffractometer equipped with graphitemonochromated Mo $K_{\alpha}$  radiation ( $\lambda = 0.71073$  Å). A preliminary orientation matrix and cell parameters were determined from three sets of  $\omega$  scans at different starting angles. Data frames were obtained at scan intervals of  $0.5^{\circ}$  with an exposure time of 10 s frame<sup>-1</sup>. The reflection data were corrected for Lorentz and polarization factors. Absorption corrections were carried out using SADABS. The structures were solved by direct methods and refined by full-matrix least-squares analysis using anisotropic thermal parameters for non-H atoms with the SHELXTL program [11]. All H atoms were calculated at idealized positions and refined with the riding models. Crystallographic data for the complexes are summarized in Table 1.

Supplementary material has been deposited with the Cambridge Crystallographic Data Centre (nos. 950909 (I) and 950910 (II); deposit@ ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

Antibacterial activity test. The in vitro activity test was carried out using the growth inhibitory zone (well method) [12]. The potency of components was determined against the Gram-positive bacteria: S. agalactiae and S. aureus and the Gram-negative bacteria: K. pneumoniae and P. aeruginosa. Microorganisms were cultured on Muller-Hinton agar medium. The inhibitory activity was compared with that of standard antibiotics, such as gentamicine (10  $\mu$ g). After drilling wells on medium using a 6 mm cork borer, 100 µL of solution from different compounds were poured into each well. The plates were incubated at 37°C overnight. The diameter of the inhibition zone was measured to the nearest. Each test was carried out in triplicate and the average was calculated for inhibition zone diameters. A blank containing only methanol showed no inhibition in a preliminary test. The macro-dilution broth susceptibility assay was used for the evaluation of minimal inhibitory concentration (MIC). By including 1 mL Muller–Hinton broth in each test, and then adding 1 mL extract with concentration 100 mg/mL in the first tube, we made serial dilution of this extract from first tube to last tube. Bacterial suspension prepared to match the turbidity of 0.5 Mcfarland turbidity standards. Matching this turbidity provides a bacterial inoculum concentration of  $1.5 \times 10^8$  cfu/mL. Then 1 mL of bacterial suspension was added to each test tube. After incubation at  $37^{\circ}$ C for 24 h, the last tube was determined as the minimal inhibitory concentration (MIC) without turbidity.

### **RESULTS AND DISCUSSION**

Two new mononuclear iron(III) complexes were synthesized by reaction of Schiff bases, acetohydroxamic acid, with iron perchlorate in methanol. For complex I, acetohydroxamic acid was coordinated to the Fe atom, while for complex II, even though we had added the material, it has not participated in coordination.

The asymmetric unit of compound I contains a mononuclear iron complex molecule and a water molecule of crystallization (Fig. 1a). The Fe atom in the complex shows distorted octahedral coordination geometry, with O(1), N(2) atoms of the Schiff base ligand, and O(5) and O(6) atoms of the AHA ligand defining the equatorial coordination sites, and N(1)and O(2) atoms of the Schiff base ligand occupying the axial positions. The Fe atom deviates from the best coordination plane defined by the equatorial atoms by 0.145(1) Å in direction of O(2) atom. The coordinate bond lengths and angles (Table 2) are in line with the corresponding values found in related six-coordinated iron(III) complexes with Schiff bases [13, 14]. The distortion of the octahedral coordination may be caused by the strain created by the five-membered chelate rings Fe(1)-N(1)-C(8)-C(9)-N(2) and Fe(1)-O(5)-C(22)-N(3)-O(6). The three trans angles are much deviated from the ideal value of 180°, ranging from  $155.9(1)^{\circ}$  to  $161.7(1)^{\circ}$ . The remaining bond angles are also deviated from the ideal value of 90°, ranging from 74.4(1)° to  $112.4(1)^\circ$ . The dihedral angle between the two benzene rings of the Schiff base ligand is  $33.5(3)^{\circ}$ .

In the crystal structure of complex I, the iron complex molecules and the water molecules are linked together by  $O-H\cdots O$  and  $N-H\cdots O$  hydrogen bonds (Table 3), to form 2D layers parallel to the *yz* plane (Fig. 2a).

The asymmetric unit of compound **II** contains two mononuclear iron complex cations and two perchlorate anions (Fig. 1b). The Fe atom in the complex shows distorted octahedral coordination geometry, with the three donor atoms of one Schiff base ligand and the imino N atom of the other Schiff base ligand

	Value		
Parameter	Ι	II	
Formula weight	516.35	537.80	
Crystal size, mm	$0.30 \times 0.27 \times 0.27$	$0.30 \times 0.27 \times 0.27$	
Temperature, K	298(2)	298(2)	
Crystal system	Orthorhombic	Triclinic	
Space group	Iba2	PĪ	
a, Å	20.781(3)	12.748(1)	
b, Å	23.527(3)	13.491(1)	
<i>c</i> , Å	10.071(2)	19.007(1)	
α, deg	90	106.623(2)	
β, deg	90	97.462(2)	
γ, deg	90	112.543(2)	
<i>V</i> , Å <sup>3</sup>	4923.7(12)	2784.9(4)	
Ζ	8	4	
$\rho_{calcd},  mg  cm^{-3}$	1.393	1.283	
$\mu(\mathrm{Mo}K_{\alpha}),\mathrm{mm}^{-1}$	0.660	0.677	
F(000)	2168	1124	
Number of measured reflections	24588	26141	
Number of observations $(I > 2\sigma(I))$	5071	10319	
Unique reflections	4132	5591	
Parameters	320	616	
Number of restraints	5	48	

0.0374, 0.0900

0.0534, 0.0999

1.043

0.280, -0.271

0.0892, 0.2434

0.1632, 0.2942

1.020

0.799, -0.572

\*  $R_1 = \sum ||F_0| - |F_c|| / \sum |F_0|, wR_2 = [\sum w (F_0^2 - 2F_c^2)^2 / \sum w (F_0^2)^2]^{1/2}.$ 

 $R_1, wR_2(I > 2\sigma(I))^*$ 

 $R_1$ ,  $wR_2$  (all data)\*

Goodness of fit of  $F^2$ 

Largest difference peak and hole,  $e \text{ Å}^{-3}$ 

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Fig. 1. ORTEP diagram of complexes I (a) and II (b) with 30% thermal ellipsoids for all non-hydrogen atoms.

defining the equatorial coordination sites, and the remaining two donor atoms of the second Schiff base ligand occupying the axial positions. The Fe(1) and Fe(2) atoms deviate from the best coordination planes defined by the equatorial atoms by 0.095(1) Å and 0.116(1) Å, respectively. The coordinate bond lengths and angles (Table 2) are comparable to those observed in complex I, and also in line with the corresponding

values found in related six-coordinated iron(III) complexes with Schiff bases [15, 16]. The distortion of the octahedral coordination may be caused by the strain created by the five-membered chelate rings Fe(1)-N(1)-C(9)-C(10)-N(2), Fe(1) - N(3) -C(20)-C(21)-N(4), Fe(2)-N(5)-C(31)-C(2)-N(6), and Fe(2)-N(7)-C(42)-C(43)-N(8). The three trans angles are much deviated from the ideal value of  $180^{\circ}$ , ranging from  $164.7(2)^{\circ}$  to  $170.4(2)^{\circ}$  for Fe(1) and from  $162.7(2)^{\circ}$  to  $168.9(2)^{\circ}$  for Fe(2). The remaining bond angles are also deviated from the ideal value of 90°, ranging from  $78.5(2)^{\circ}$  to  $100.1(2)^{\circ}$  for Fe(1) and from  $77.8(2)^{\circ}$  to  $100.8(2)^{\circ}$  for Fe(2). The two Schiff base ligands in the complex are also vertical to each other, with dihedral angles of  $99.6(3)^{\circ}$  for Fe(1) complex molecule, and  $108.0(3)^{\circ}$  for Fe(2) complex molecule.

In the crystal structure of complex II, the complex cations and the perchlorate anions are linked together by N-H...O hydrogen bonds (Table 3), to form dimers (Fig. 2b).

The infrared spectra of the Schiff bases and their complexes provide information about the metalligand bonding. The medium and broad absorptions in the 3200–3500 cm<sup>-1</sup> region for the Schiff bases and complex I substantiate the presence of O-H groups. The sharp and weak absorptions at 3227 and 3258 cm<sup>-1</sup> for complexes I and II are assigned to N-H groups. Strong absorptions at 1645  $cm^{-1}$  for  $H_2L^1$  and  $1639 \text{ cm}^{-1}$  for HL<sup>2</sup> are assigned to the azomethine groups, v(C=N) [17]. The bands appeared at  $1633 \text{ cm}^{-1}$  for I and  $1636 \text{ cm}^{-1}$  for II, which can be attributed to donation of the nitrogen atom lone pair of the azomethine groups to the metal ions. The phenolic v(C-O) in the free Schiff base ligands exhibit strong bands at 1262 cm<sup>-1</sup> for H<sub>2</sub>L<sup>1</sup> and 1253 cm<sup>-1</sup> for HL<sup>2</sup>. In the complexes, the bands appear at lower wave numbers, viz. 1243 cm<sup>-1</sup> for I and 1235 cm<sup>-1</sup> for II, when compared to those of the free Schiff bases. In the spectrum of II, the intense band centered at  $1115 \text{ cm}^{-1}$ is attributed to the perchlorate anions. Conclusive evidence of the bonding is also shown by the presence of new bands in the spectra of the complexes at about 520-570 and 410-490 cm<sup>-1</sup>, which can be attributed to v(Fe-O) and v(Fe-N) stretching vibrations, respectively [18].

TG-DTA curves of complex I are shown in Fig. 3a. The mass loss corresponding to one lattice water molecule is observed at  $100-170^{\circ}$ C. The organic part of the complex decomposes into roughly two steps, the first lies in the range  $170-218^{\circ}$ C with a DTG peak at 222°C. The mass loss (14.0%) corresponds to loss of the AHA ligand. The next stage of decomposition starts at 220°C and ends at 450°C. Since the TG curve of the complex showed continuous degradation at this stage, we were not able to isolate the intermediates. The steady mass loss observed at this stage may be due

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

Bond	<i>d</i> , Å	Bond	<i>d</i> , Å		
	I				
Fe(1)-O(1)	1.969(2)	Fe(1)–O(2)	1.933(2)		
Fe(1)-O(5)	2.084(2)	Fe(1)–O(6)	1.9657(18)		
Fe(1)-N(1)	2.113(3)	Fe(1)-N(2)	2.189(3)		
	j	Ĩ			
Fe(1)-O(1)	1.881(5)	Fe(1)–O(2)	1.888(5)		
Fe(1)-N(1)	2.143(5)	Fe(1)–N(2)	2.176(6)		
Fe(1)-N(3)	2.133(5)	Fe(1)–N(4)	2.148(6)		
Fe(2)-O(3)	1.899(5)	Fe(2)–O(4)	1.882(5)		
Fe(2)-N(5)	2.136(5)	Fe(2)–N(6)	2.161(6)		
Fe(2)-N(7)	2.144(5)	Fe(2)–N(8)	2.163(5)		
Angle	ω, deg	Angle	ω, deg		
	-	I			
O(2)Fe(1)O(6)	103.3(1)	O(2)Fe(1)O(1)	94.0(1)		
O(6)Fe(1)O(1)	86.0(1)	O(2)Fe(1)O(5)	98.4(1)		
O(6)Fe(1)O(5)	78.2(1)	O(1)Fe(1)O(5)	161.7(1)		
O(2)Fe(1)N(1)	155.9(1)	O(6)Fe(1)N(1)	100.6(1)		
O(1)Fe(1)N(1)	84.6(1)	O(5)Fe(1)N(1)	89.4(1)		
O(2)Fe(1)N(2)	84.1(1)	O(6)Fe(1)N(2)	159.9(1)		
O(1)Fe(1)N(2)	112.4(1)	O(5)Fe(1)N(2)	82.3(1)		
N(1)Fe(1)N(2)	74.4(1)				
Π					
O(1)Fe(1)O(2)	96.2(3)	O(1)Fe(1)N(3)	100.1(2)		
O(2)Fe(1)N(3)	86.7(2)	O(1)Fe(1)N(1)	86.3(2)		
O(2)Fe(1)N(1)	99.8(2)	N(3)Fe(1)N(1)	170.4(2)		
O(1)Fe(1)N(4)	90.4(2)	O(2)Fe(1)N(4)	164.7(2)		
N(3)Fe(1)N(4)	78.5(2)	N(1)Fe(1)N(4)	94.3(2)		
O(1)Fe(1)N(2)	165.6(2)	O(2)Fe(1)N(2)	88.8(2)		
N(3)Fe(1)N(2)	93.7(2)	N(1)Fe(1)N(2)	79.5(2)		
N(4)Fe(1)N(2)	88.2(2)	O(4)Fe(2)O(3)	96.7(3)		
O(4)Fe(2)N(5)	100.8(2)	O(3)Fe(2)N(5)	85.2(2)		
O(4)Fe(2)N(7)	86.2(2)	O(3)Fe(2)N(7)	102.7(2)		
N(5)Fe(2)N(7)	168.9(2)	O(4)Fe(2)N(6)	89.3(3)		
O(3)Fe(2)N(6)	162.7(2)	N(5)Fe(2)N(6)	77.8(2)		
N(7)Fe(2)N(6)	93.8(2)	O(4)Fe(2)N(8)	164.6(2)		
O(3)Fe(2)N(8)	89.8(2)	N(5)Fe(2)N(8)	93.5(2)		
N(7)Fe(2)N(8)	78.8(2)	N(6)Fe(2)N(8)	88.4(2)		

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D–H…A					
	D-H	Н…А	D…A	Angle DHA, deg	
O(7)-H(7B)····O(3) <sup>i</sup>	0.85(1)	2.31(2)	3.026(4)	144(3)	
$O(7) - H(7B) - O(1)^{i}$	0.85(1)	2.31(2)	3.044(3)	147(3)	
N(3)-H(3)-0(7) <sup>ii</sup>	0.90(1)	1.92(1)	2.810(4)	171(4)	
I II					
N(8)-H(8B)…O(6)	0.90	2.41	3.28(2)	160	
N(8)-H(8A)····O(11) <sup>iii</sup>	0.90	2.16	3.04(1)	167	
N(6)-H(6B)····O(9) <sup>iv</sup>	0.90	2.37	3.15(1)	146	
N(6)-H(6A)O(9) <sup>iii</sup>	0.90	2.35	3.21(2)	158	
$N(4)-H(4B)\cdots O(8)^{iii}$	0.90	2.31	3.06(1)	140	
N(4)-H(4A)····O(8)	0.90	2.34	3.23(1)	168	
N(2)-H(2A)····O(6)	0.90	2.52	3.27(2)	142	
N(2)-H(2A)···O(7)	0.90	2.44	3.16(1)	138	

Table 3. Geometric parameters of hydrogen bonds for I and  $II^*$ 

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

to the removal of the Schiff base ligand, and the formation of final product ( $Fe_2O_3$ ).

TG–DTA curves of complex II are shown in Fig. 3b. The mass loss corresponding to one perchlorate anion is observed at  $110-160^{\circ}$ C. The organic part of the complex decomposes into roughly two steps, the first lies in the range 208–240°C with a DTG peak at 218°C. The mass loss (50.0%) corresponds to loss of the four outer atoms of the benzene rings and the methyl atoms of the Schiff base ligands. The next stage of decomposition starts at 240°C and ends at 460°C. The steady mass loss observed at this stage may be due to the removal of the remaining parts of the Schiff base ligands, and the formation of final product (Fe<sub>2</sub>O<sub>3</sub>).

Antibacterial activities of the Schiff bases and the two complexes as well as the reference drug gentamicine are summarized in Table 4 (zone of growth inhibition and minimal inhibitory concentrations). The results indicate strong activity of the free Schiff bases against Gram-positive bacteria, *S. agalactiae*, and moderate activities of them toward Gram-positive bacteria *S. aureus* and Gram-negative bacteria *K. pneumoniae* and *P. aeruginosa*. In comparison, the iron complexes have stronger activities against all the bacteria than the free Schiff bases. The increased activity of the complexes may be explained on the basis of chelation theory; chelation reduces the polarity of the metal atom mainly because of partial sharing of its positive charge with the donor groups and possible

Gram-positive and -negative bacteria	Gentamicine	$H_2L^1$	HL <sup>2</sup>	Ι	П
S. agalactiae		20 (25)	10 (50)	30 (6.25)	30 (12.5)
S. aureus	20	10 (25)	10 (25)	40 (3.125)	30 (6.25)
K. pneumoniae	20	20 (12.5)	20 (12.5)	30 (6.25)	40 (3.125)
P. aeruginosa	15	10 (50)	10 (25)	30 (12.5)	20 (12.5)

Table 4. Growth inhibition zone (mm) and MIC (mg/mL) values (in brackets) of compounds  $H_2L^1$ ,  $HL^2$ , I, and II

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(b)



Fig. 2. Molecular packing structure of complexes I (a) and II (b), viewed along the y and z axes, respectively. Hydrogen bonds are drawn as dotted lines.

*p*-electron delocalization within the whole chelation. Also, chelation increases the lipophilic nature of the central atom which subsequently favors its permeation through the lipid layer of the cell membrane [19]. The quantitative assays gave MIC values in the range  $3.125-50 \text{ mg mL}^{-1}$ , that confirmed the above obtained results.

In summary, the present paper reports the synthesis, characterization and crystal structures of two new iron(III) complexes with Schiff bases. The complexes show effective antibacterial activities against Grampositive bacteria *S. agalactiae* and *S. aureus* and Gram-negative bacteria *K. pneumoniae* and *P. aeruginosa*. Further work might be carried out to explore

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Fig. 3. TG-DTA curves of complexes I (a) and II (b).

more effective antibacterial materials based on the present complex models.

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