Synthesis, Characterization, and Chiral Assembly Structure of a Self-Complementary Iron(III) Complex, $[Fe^{III}(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})](ClO_4)_2\cdot EtOH$ ($H_2L^{5\text{-}Me}$: *N*-[(5-Methylimidazol-4-yl)methylidene]histamine)

Hiroaki Hagiwara,[1] Koshiro Nishi,[1] Naohide Matsumoto,*[1] Yukinari Sunatsuki,[2] Masaaki Kojima,[2] and Seiichiro Iijima[3]

[1]Department of Chemistry, Graduate School of Science and Technology, Kumamoto University,
2-39-1 Kurokami, Kumamoto 860-8555

[2]Department of Chemistry, Faculty of Science, Okayama University, 3-1-1 Tsushima-naka, Okayama 700-8530

[3]National Institute of Advanced Industrial Science and Technology, Tsukuba 305-8566

Received October 25, 2010; E-mail: naohide@aster.sci.kumamoto-u.ac.jp

A low-spin Fe^{III} complex, $[Fe^{III}(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})](ClO_4)_2\cdot EtOH$, was prepared by the reaction of $Fe^{II}(ClO_4)_2\cdot 6H_2O$ and $H_2L^{5\text{-}Me}$ in ethanol with 1:2 molar ratio and in air, where $H_2L^{5\text{-}Me}$: *N*-[(5-methylimidazol-4-yl)methylidene]histamine. Each complex molecule of $[Fe(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})]^{2+}$ has both fully protonated ligand and mono-deprotonated ligand and acts as a chiral self-complementary building block to produce the homochiral imidazole–imidazolate-bridged zigzag-chain structure. The X-ray analysis confirmed that the adjacent chains with the same chirality are stacked in a crystal lattice to give a chiral crystal (conglomerate). Low-spin Fe^{III} was revealed by the magnetic susceptibility and Mössbauer measurements.

Metal complexes of polydentate imidazole containing ligands are useful complementary building components for construction of polynuclear clusters and multidimensional architecture by coordination or hydrogen bonds.[1] Furthermore, metal complexes of imidazole-containing ligand, especially iron complexes, provide versatile oxidation and spin states with or without various spin transitions depending on the ligand frameworks, assembly structures, and the protonation degree of the imidazole moiety.[2]

Tadokoro et al.[3] and Haga et al.[4] have reported that metal complexes containing imidazole groups can show acid–base-promoted redox processes and spin state selection with the formation of complementary intermolecular hydrogen bonds. We have studied the iron complexes of a tripodal ligand containing three imidazole groups, H_3L: tris(2-{[(imidazole-4-yl)methylidene]amino}ethyl)amine or H_3L^{Me}: tris(2-{[(2-methylimidazole-4-yl)methylidene]amino}ethyl)amine, and synthesized a series of iron complexes, including fully deprotonated Fe^{III} complex, half deprotonated both Fe^{II}–Fe^{III} mixed-valence and Fe^{II}–Fe^{II} complex with averaged or proton-ordered 2D imidazole–imidazolate hydrogen-bond networks, and fully protonated Fe^{II} complex, which showed various electronic states and spin crossover (SCO) properties by the adjustment of the protonation degree.[5]

Recently, we have also reported the Fe^{II} complexes of unsymmetrical tridentate ligands with N_3 donor atoms derived from the 1:1 condensation reaction of 2-methyl-4-formylimidazole and histamine, $H_2L^{2\text{-}Me}$: *N*-[(2-methylimidazol-4-yl)methylidene]histamine.[6] A variety of SCO Fe^{II} complexes with the protonated ligand were observed, demonstrating that the modification of the tridentate ligand system can produce a rich chemistry in the control of the oxidation and spin state as well as the network structure.

In this work, we have focused on iron complexes with an analogous tridentate ligand, $H_2L^{5\text{-}Me}$: *N*-[(5-methylimidazol-4-yl)methylidene]histamine, which show donor–acceptor ability as a self-complementary building block by deprotonation of the ligand. On the basis of previous work, we know that ligands prepared by the condensation of 4-methyl-5-formylimidazole and amine derivatives generally gives a stronger ligand field than analogous ligands using 2-methyl-4-formylimidazole.[7] So, to generate versatile oxidation and spin states correlating with the protonation degree of the imidazole moiety, a stronger ligand $H_2L^{5\text{-}Me}$ is preferable to $H_2L^{2\text{-}Me}$ (Chart 1). Now, we synthesized an imidazole–imidazolate-bridged 1D iron(III) complex from an iron(II) metal source in air without the adjustment of the pH. Here we report a low-spin (LS) Fe^{III} complex with homochiral 1D zigzag-chain structure constructed by imidazole–imidazolate ($NH\cdots N^-$) hydrogen bonds of self-complementary chiral building block, $[Fe(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})](ClO_4)_2\cdot EtOH$ (**1**) ($H_2L^{5\text{-}Me}$: fully protonated tridentate N_3 ligand *N*-[(5-methylimidazol-4-yl)methylidene]histamine and $HL^{5\text{-}Me}$: mono-deprotonated type of $H_2L^{5\text{-}Me}$).

Results and Discussion

The synthesis of **1** was performed in air. The tridentate ligand, $H_2L^{5\text{-}Me}$, was prepared by mixing 4-methyl-5-formylimidazole and histamine in a 1:1 molar ratio in ethanol. To the

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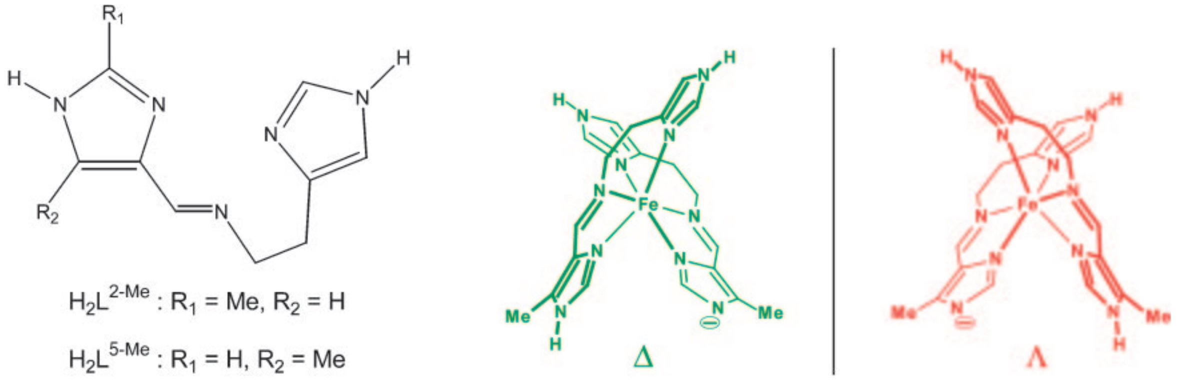

Chart 1. Schematic drawing of the ligand: H_2L^R and two possible enantiomorphs of mono-deprotonated complex $[Fe(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})]^{2+}$.

ligand solution was added an ethanol solution of $Fe^{II}(ClO_4)_2 \cdot 6H_2O$. The color of the solution changed from colorless to dark-red when the solution of $Fe^{II}(ClO_4)_2 \cdot 6H_2O$ was added to the ligand solution. The resulting solution was allowed to stand for several days to produce plate-like dark-red crystals. The C, H, and N elemental analyses agree with the chemical formula of $[Fe^{III}(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})](ClO_4)_2 \cdot EtOH$. The oxidation state of metal ion was confirmed later to be iron(III) by physical measurements including temperature-dependence magnetic susceptibility, Mössbauer spectra, and X-ray diffraction analysis. One EtOH molecule as the crystal solvent was detected by thermogravimetric analyses (TGA), where a small amount of sample (ca. 2 mg) was used for the TGA measurement due to the potential explosiveness of the perchlorate salt.

The crystal structure of **1** was determined by single-crystal X-ray analysis at 110 K. The complex **1** crystallized in the monoclinic acentrosymmetric space group $P2_1$ (No. 4) with $Z = 2$. The structure consists of one $[Fe(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})]^{2+}$, two ClO_4^- ions, and an ethanol molecule as a crystal solvent. The molecular structure of $[Fe(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})](ClO_4)_2 \cdot EtOH$ (**1**) with selected atoms numbered is shown in Figure 1. The selected bond lengths and angles are given in Table 1. Hydrogen bond distances and angles are given in Table 2. The Fe ion has an octahedral coordination environment with N_6 donor atoms of two unsymmetrical tridentate Schiff-base ligands and the complex cation is a chiral species with Δ- or Λ-isomer (Chart 1).

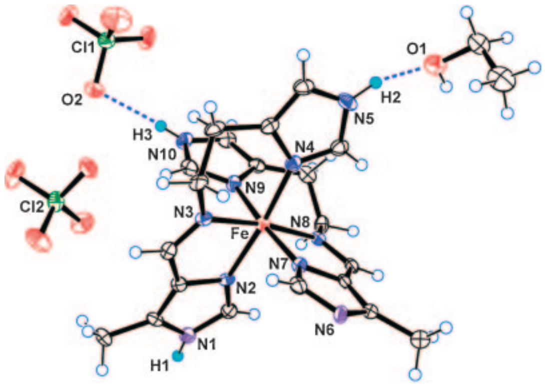

Figure 1. Molecular structure of $[Fe(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})](ClO_4)_2 \cdot EtOH$ (**1**) at 110 K with selected atoms numbered, where the thermal ellipsoids are drawn with a 50% probability level. Although the complex cation has essentially four imidazole NH sites at the terminal sites, only the imidazole N(6) atom in 4-methyl-5-formylimidazole is deprotonated. The complex cation is chiral due to octahedral coordination of two unsymmetrical tridentate ligands. The nitrogen atom N(5) of the imidazole moiety of the complex cation is hydrogen-bonded to an ethanol molecule and the nitrogen atom N(10) of the imidazole moiety is hydrogen-bonded to a perchlorate anion $Cl(1)O_4^-$. As a result, two imidazole groups N(5)H and N(10)H are blocked and further contact is prevented. Another perchlorate anion $Cl(2)O_4^-$ exists as an isolated ion.

There are four imidazole groups per metal complex, in which three hydrogen atoms bonded to imidazole nitrogen atoms were located on a difference Fourier map, but a peak assignable to the hydrogen bonded to the N(6) atom at one of two 4-methyl-5-formylimidazole moieties could not be found. The X-ray diffraction indicates that one tridentate ligand is fully protonated and the other is mono-deprotonated. This also suggests that the hydrogen atom from histamine is harder to remove from the imidazole nitrogen atom than that of 4-methyl-5-formylimidazole. On the basis of the three imidazole groups and one imidazolate group per metal complex described above, the chemical formula of $[Fe(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})]$ has been postulated. Considering the complex involves two perchlorate anion, the chemical formula of dipositive complex-cation with $[Fe(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})]^{2+}$ is postulated. The Fe–N bond distances are in the range of 1.914(4) and 1.971(4) Å, and the average Fe–N distance is 1.953 Å, consistent with the values of the LS Fe^{III} state, demonstrating that the oxidation state of iron(II) is oxidized to iron(III) in air. Among the three Fe–N distances of tridentate ligand, the Fe–N distance at the formylimidazole moiety is the shortest. The Fe–N distances (Fe–N(2) = 1.936(4) Å) for the protonated imidazole moiety is longer than that (Fe–N(7) = 1.914(4) Å) for the deprotonated imidazolate moiety.

The nitrogen atom N(5) of the imidazole moiety of the complex cation is hydrogen-bonded to an ethanol molecule with the distance of N(5)···O(1) = 2.764(4) Å and the nitrogen atom N(10) of the imidazole moiety of the complex cation is hydrogen-bonded to a perchlorate anion $Cl(1)O_4^-$ with the distance of N(10)···O(2) = 2.960(6) Å, in which both N(5) and

Table 1. Relevant Bond Lengths (Å) and Angles (degree) for $[Fe(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})](ClO_4)_2 \cdot EtOH$ (**1**) at 110 K

Bond lengths/Å	
Fe–N(2)	1.936(4)
Fe–N(3)	1.968(3)
Fe–N(4)	1.957(4)
Fe–N(7)	1.914(4)
Fe–N(8)	1.969(3)
Fe–N(9)	1.971(4)
Bond angles/degree	
N(2)–Fe–N(3)	81.94(14)
N(2)–Fe–N(4)	173.84(14)
N(2)–Fe–N(7)	91.95(15)
N(2)–Fe–N(8)	92.44(14)
N(2)–Fe–N(9)	90.25(15)
N(3)–Fe–N(4)	92.46(15)
N(3)–Fe–N(7)	92.66(14)
N(3)–Fe–N(8)	172.48(17)
N(3)–Fe–N(9)	93.58(13)
N(4)–Fe–N(7)	90.88(15)
N(4)–Fe–N(8)	93.36(14)
N(4)–Fe–N(9)	87.51(15)
N(7)–Fe–N(8)	82.50(14)
N(7)–Fe–N(9)	173.62(12)
N(8)–Fe–N(9)	91.43(14)

Table 2. Hydrogen Bond Distances (Å) and Angles (degree) for $[Fe(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})](ClO_4)_2 \cdot EtOH$ (**1**) at 110 K

Donor	H	Acceptor	D···A	D–H	H···A	D–H···A
N(1)	H(1)	N(6)* a)	2.743(5)	1.02	1.74(5)	167(4)
N(5)	H(2)	O(1)	2.764(4)	0.93	1.88(4)	158(5)
N(10)	H(3)	O(2)	2.960(6)	0.81	2.17(5)	164(4)

a) Symmetry operations: $-x + 1, y - 1/2, -z$.

N(10) atoms originate from histamine. As a result, two imidazole groups N(5)H and N(10)H per molecule are blocked and further contact is prevented. Another perchlorate anion $Cl(2)O_4{}^-$ exists as an isolated ion.

As shown in Figure 2, adjacent complex cations are connected by NH···N⁻ hydrogen bonds between their imidazole N(1)H and imidazolate N(6)* moieties with the distance of N(1)···N(6)* = 2.743(5) Å (*: $-x + 1, y - 1/2, -z$) and form a 1D zigzag-chain structure. Within a chain, the complex cations with the same chirality are linked by NH···N⁻ hydrogen bonds. It should be noted that the present NH···N⁻ hydrogen bond is constructed by only the imidazole and imidazolate sites from 4-methyl-5-formylimidazole. In the formation of the homochiral 1D zigzag-chain structure, each complex molecule of $[Fe(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})]^{2+}$ has both donor and acceptor abilities for the formation of the intermolecular hydrogen bond, which prefers a proton-ordered state to a proton-disordered state, and acts as a chiral self-complementary building block. Previously, various metal complexes with both proton-located[8] and averaged proton-unlocated[9] intermolecular imidazole–imidazolate hydrogen bonding in the crystalline state have been reported. The present compound was determined to be proton-located.

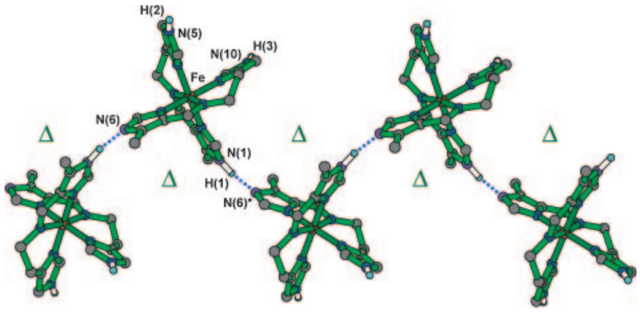

Figure 2. Intermolecular interactions of $[Fe(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})](ClO_4)_2 \cdot EtOH$ (**1**) at 110 K. Adjacent complex cations are connected by NH···N⁻ hydrogen bonds between their imidazole N(1)H and imidazolate N(6)* moieties (*: $-x + 1, y - 1/2, -z$).

As shown in Figure 3a, homochiral 1D zigzag-chain formed by NH···N⁻ hydrogen bonds runs along the *b* axis. The adjacent 1D chains having the same chirality are stacked in a crystal lattice resulting a chiral crystal (conglomerate), as shown in Figures 3a and 3b. Analogous Fe^{II} complexes with unsymmetrical tridentate ligands of 1:1 condensation product of 2-methyl-4-formylimidazole and histamine, $H_2L^{2\text{-}Me}$: *N*-[(2-methylimidazol-4-yl)methylidene]histamine, have homochiral 1D network structure formed by doubly bridged NH···Cl⁻ hydrogen bonds.[6] These results suggest that this type of unsymmetrical tridentate ligand is suitable for constructing homochiral 1D networks.

The magnetic susceptibilities of the microcrystalline samples of **1** were measured in the temperature range of 5–300 K at $1\,K\,min^{-1}$ sweeping mode under an applied magnetic field of 0.5 T. The $\chi_M T$ versus T plots are shown in Figure 4. The $\chi_M T$ value of **1** is constant in the 300–5 K temperature range at ca. $0.5\,cm^3\,K\,mol^{-1}$, indicating that **1** is a LS Fe^{III} complex ($S = 1/2$).

The Mössbauer spectra of **1** measured at 78 and 298 K are shown in Figure 5. Table 3 lists the Mössbauer parameters at each temperature. As shown in Figure 5, each spectrum consists only of a doublet attributable to the LS Fe^{III} species (at 78 K, isomer shift $\delta = 0.15\,mm\,s^{-1}$, quadrupole splitting $\Delta E_Q = 2.80\,mm\,s^{-1}$, at 298 K, isomer shift $\delta = 0.07\,mm\,s^{-1}$, quadrupole splitting $\Delta E_Q = 2.74\,mm\,s^{-1}$). Therefore, the Mössbauer spectra are consistent with the single-crystal X-ray analysis and the magnetic susceptibility. It should be noted that the mono-deprotonated LS Fe^{III} complex was obtained from Fe^{II} salt with no pH control in air. The Mössbauer spectrum at each temperature exhibited asymmetry where the low energy peak is fairly broader than that at high energy. This behavior is ascribed to slow paramagnetic relaxation resulting from the effective correlation time of spin relaxation on a Mössbauer time scale (10^{-7} s).[10] The further broadening at lower temperature (78 K) is reasonable in the case of slow paramagnetic relaxation. The effect of slow paramagnetic relaxation is often observable in the Mössbauer spectra of LS Fe^{III} complexes. In particular, when the Fe ions in the crystal lattice are distanced typically $\gtrsim 8$ Å from each other, distinct asymmetric line-broadenings are reported.[10b,11] The shortest Fe–Fe distance in **1** is, in fact, 8.418 Å (110 K). On the other hand, the shortest Fe–Fe distance in the related complex $[Fe^{III}L^{Me}] \cdot 3.5H_2O$ is

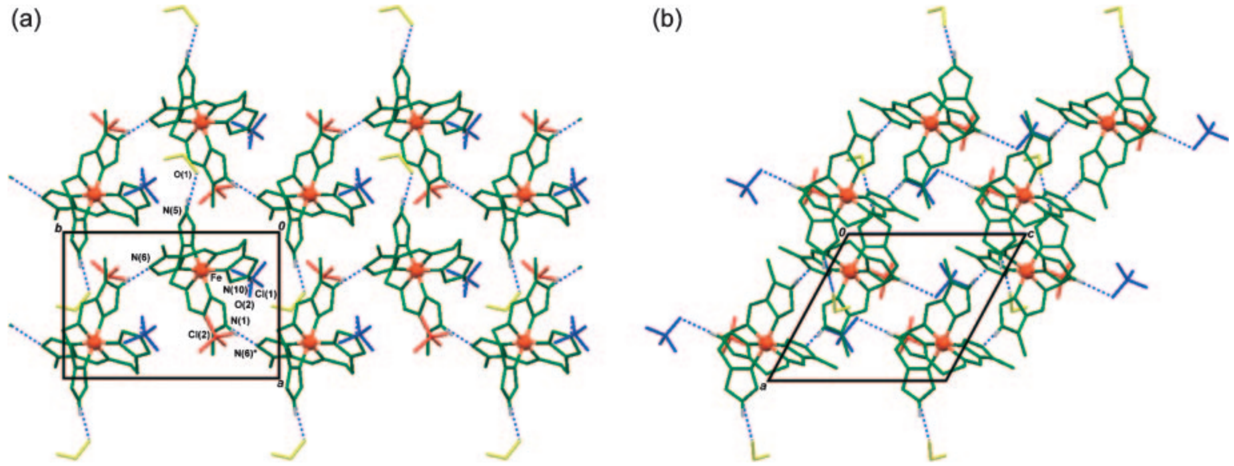

Figure 3. (a) Homochiral 1D zigzag-chain structure of $[Fe(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})](ClO_4)_2 \cdot EtOH$ (**1**) at 110 K formed by $NH \cdots N^-$ hydrogen bonds between their imidazole N(1)H and imidazolate N(6)* moieties. Adjacent 1D chains with the same chirality running along the *b* axis are stacked along the *a* axis. (b) Adjacent 1D chains with the same chirality are also stacked along the *c* axis to give a chiral crystal (conglomerate).

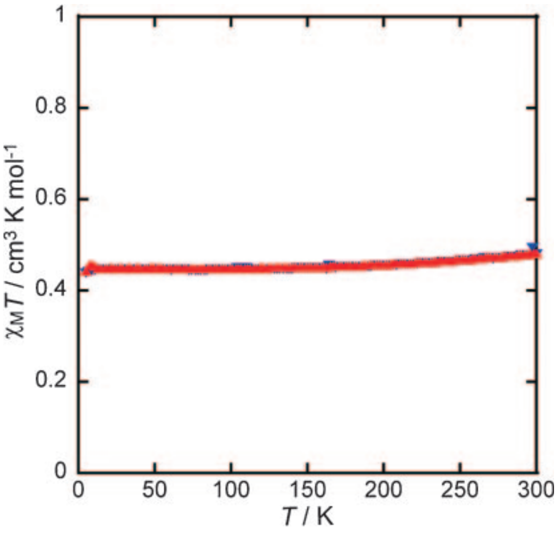

Figure 4. Magnetic behavior of $[Fe(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})](ClO_4)_2 \cdot EtOH$ (**1**) in the form of $\chi_M T$ versus T plots. The sample was cooled from 300 to 5 K (▼) and then warmed from 5 to 300 K (▲) at a sweep rate of 1 K min^{-1}.

Table 3. Mössbauer Data for $[Fe(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})](ClO_4)_2 \cdot EtOH$ (**1**)

Temp/K	$\delta^{a)}$/mm s^{-1}	ΔE_Q/mm s^{-1}	$\Gamma^{b)}$/mm s^{-1}	Assignment
298	0.07	2.74	0.42, 0.32	LS Fe^{III}
78	0.15	2.80	0.58, 0.35	LS Fe^{III}

a) Isomer shift data are reported to iron foil. b) Full width at half-height.

7.355 Å (293 K), and this complex exhibits an usual symmetric quadrupole doublet in low-spin state,[5b] where L^{Me} is fully deprotonated tris(2-{[(2-metylimidazol-4-yl)methylidene]amino}ethyl)amine (H_3L^{Me}).

Conclusion

In summary, a mono-deprotonated LS Fe^{III} complex, $[Fe^{III}(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})](ClO_4)_2 \cdot EtOH$ (**1**), was synthesized from Fe^{II} salt with no pH control in air, where $H_2L^{5\text{-}Me}$: unsymmetrical tridentate N_3 Schiff-base ligand of the 1:1 condensation product of 4-methyl-5-formylimidazole and histamine. Each complex molecule of $[Fe(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})]^{2+}$ has both donor and acceptor abilities for the formation of the intermolecular imidazole–imidazolate hydrogen bond, which prefers the proton-ordered state to the proton-disordered state, and functions as a chiral self-complementary building block to produce homochiral 1D zigzag-chain structure. The adjacent 1D chains with the same chirality are stacked in a crystal lattice resulting a chiral crystal (conglomerate). The present result suggests that the proton of the ligand $H_2L^{5\text{-}Me}$ is more acidic than that of $H_2L^{2\text{-}Me}$. In other words, the hydrogen atom on 4-methyl-5-formylimidazole of $H_2L^{5\text{-}Me}$ is easier to remove from the imidazole nitrogen atom than that of 2-methyl-4-formylimidazole of $H_2L^{2\text{-}Me}$. Hence, the present ligand system

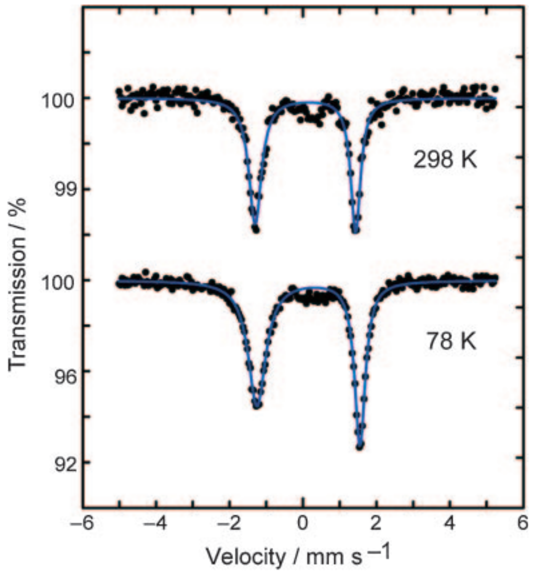

Figure 5. ^{57}Fe Mössbauer spectra of $[Fe(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})](ClO_4)_2 \cdot EtOH$ (**1**) recorded at 78 and 298 K.

induces intense air oxidation of the iron(II) metal center by increasing basicity correlated with the mono-deprotonation of the ligand. Further study on the correlation between protonation degree or proton transfer and versatile oxidation or spin states in the present ligand system is now being studied.

Experimental

Materials. All chemicals and solvents were of reagent grade, and were obtained from Tokyo Kasei Co., Ltd., and Wako Pure Chemical Industries, Ltd. They were used for syntheses without further purification.

Preparation of $[Fe(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})](ClO_4)_2\cdot EtOH$ (1). The synthesis of **1** was performed in air. Histamine dihydrochloride (0.184 g, 1.0 mmol) in 5 mL of ethanol was neutralized by 5 mL of 0.4 M NaOH ethanol solution. To the resulting mixture a solution of 4-methyl-5-formylimidazole (0.110 g, 1.0 mmol) in 5 mL of ethanol was added and the mixture was stirred for 30 min at ambient temperature. The ligand solution was used for the synthesis of the iron complex without isolating the ligands. To a solution of the ligand (1.0 mmol) was added a solution of $Fe^{II}(ClO_4)_2\cdot 6H_2O$ (0.181 g, 0.5 mmol) in 5 mL of ethanol. The color of the resulting solution changed from colorless to dark-red when the $Fe^{II}(ClO_4)_2\cdot 6H_2O$ solution was added to the ligand solution. The mixture was stirred for 5 min at ambient temperature and then filtered to remove the white-colored sodium chloride. The filtrate was left to stand for several days, during which time dark-red plate crystals precipitated. They were collected by suction filtration. Yield: 0.353 g (75%). Anal. Calcd for $[Fe(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})](ClO_4)_2\cdot EtOH = C_{22}H_{31}N_{10}O_9Cl_2Fe$: C, 37.41; H, 4.42; N, 19.83%. Found: C, 37.77; H, 4.68; N, 19.89%.

CAUTION! The perchlorate salts of metal complexes with organic ligands are potentially explosive. Only small quantities of the compound should be prepared, and they should be handled with much care!

Physical Measurements. The elemental C, H, and N analyses were carried out by Miss. Kikue Nishiyama at the Center for Instrumental Analysis of Kumamoto University. Thermogravimetric analyses (TGA) were performed on a TG/DTA6200 (Seiko Instrument Inc.), where a sample of ca. 2 mg was heated from room temperature to 130 °C. Due to the potential explosion of the ClO_4 salt, the measurement was performed by the use of a small amount of sample at lower than 130 °C. Magnetic susceptibilities were measured using a MPMS5 SQUID susceptometer (Quantum Design) in the 5–300 K temperature range under an applied magnetic field of 0.5 T. The calibration was done with palladium metal. Corrections for diamagnetism were applied using Pascal's constants.[12] Mössbauer spectra were recorded using a Wissel 1200 spectrometer and a proportional counter. $^{57}Co(Rh)$ moving in a constant acceleration mode was used as the radioactive source. Hyperfine parameters were obtained by a least-squares fitting of the Lorentzian peaks. Isomer shifts are reported relative to iron foil at 293 K. The sample temperature was controlled by means of a Heli-tran liquid transfer refrigerator (Air Products and Chemicals, Inc.) to within an accuracy of ±0.5 K.

X-ray Crystallography. A dark-red plate crystal having approximate dimensions of $0.39 \times 0.26 \times 0.07\ \mathrm{mm}^3$ was mounted on a glass fiber. The X-ray diffraction data were collected by a Rigaku RAXIS RAPID imaging plate diffractometer using graphite monochromated Mo Kα radiation ($\lambda = 0.71075$ Å). The temperature of the crystal was maintained at 110 K by means of a Rigaku cooling device to within an accuracy of ±1 K. The data were corrected for Lorentz, polarization, and absorption effects. The structure was solved by direct method and expanded using Fourier technique. The structures were refined on F^2 full-matrix least-squares with anisotropic displacement parameters for all non-hydrogen atoms. The positions of all hydrogen atoms were successfully located on the difference Fourier map. The possible hydrogen atom, which might bond to the N(6) atom from 4-methyl-5-formylimidazole could not be found. It was experimentally confirmed that the N(6) is the imidazolate group. Finally, all hydrogen atoms were refined isotropically. All calculations were performed using the Crystal Structure crystallographic software package.[13] Flack parameter analysis suggested that the measured crystal was chiral with Δ form enantiomer.

X-ray crystallographic data for $[Fe(H_2L^{5\text{-}Me})(HL^{5\text{-}Me})](ClO_4)_2\cdot EtOH$ at 110 K: formula: $C_{22}H_{31}N_{10}O_9Cl_2Fe$, fw: 706.30, monoclinic, space group: $P2_1$ (No. 4), $a = 10.7872(5)$, $b = 13.7407(6)$, $c = 11.2993(6)$ Å, $\beta = 118.669(2)°$, $V = 1469.5(2)$ Å^3, $Z = 2$, $D_{\mathrm{calcd}} = 1.596\ \mathrm{g\,cm^{-3}}$, $\mu(\mathrm{Mo\,K}\alpha) = 7.623\ \mathrm{cm^{-1}}$, R_1 $[I > 2\sigma(I)] = 0.0373$, wR_2 $[I > 2\sigma(I)] = 0.0540$, GOF = 1.019, Flack parameter for Δ form = 0.01(2). X-ray crystallographic data in CIF format has been deposited with the deposition number 800970 at CCDC. Copies of the data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge, CB2 1EZ, U.K.; Fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk).

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