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Copper(II) complexes with multidentate Schiff-base ligands containing imidazole groups: ligand-complex or self-complementary molecule?

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Contents

Abstract

The copper(II) complexes with multidentate Schiff-base ligands containing imidazole groups have potentially donor and acceptor character in the formation of a coordination bond and a hydrogen bond. They can function as a *ligand-complex* or as a self-complementary building block for the construction of the assembly structure due to the formation of a coordination bond or a hydrogen bond. In such self-complementary complexes, the monomer is stabilized as a protonated species under acidic conditions, while under appropriate basic conditions the generated imidazolate nitrogen atom coordinates to the Cu(II) ion of the adjacent unit or hydrogen-bonds to the imidazole group of the adjacent unit to give assembly structures in the crystals, depending on the ligand framework and the preferred coordination number of the Cu(II) ion. The interconversion between the monomer and the self-assembled oligomer is reversible by pH adjustment.

Keywords: Supramolecular chemistry; Hydrogen bond; Copper(II) complexes

1. Introduction

A metal complex is a treasury of functional vectors that have the diversity of geometric and electronic structures; versatile functional materials can be created by rational molecular design when the functional vector accumulates. The self-assembly process involving a metal complex is especially attractive and useful for the purpose of the construction of a variety of molecular structures and functional materials, because a metal ion together with its ligand contains not only effective structural information to guide the self-assembly reaction but it also contains various functional vectors due to its electronic structure [1–4]. Two types of building block methods using a metal complex, methods **I** and **II**, have been successful: (1) method **I**, in which one kind of metal complex contains simultaneously the donor and acceptor ability as a self-complementary molecular building block to construct the assembly

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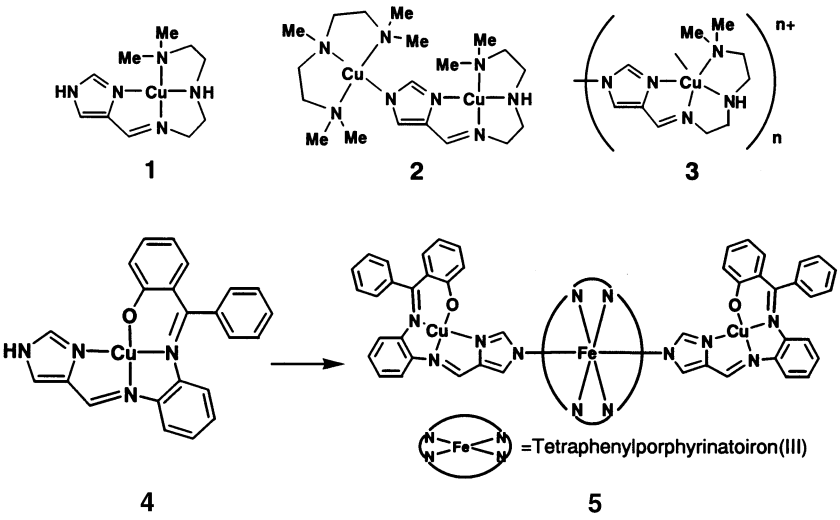
structure [5,6]; (2) method **II**, in which two kinds of metal complexes which contain donor and acceptor ability, respectively, are used as molecular building blocks. Method **II** has been very successful in creating molecular-based magnetic materials [7].

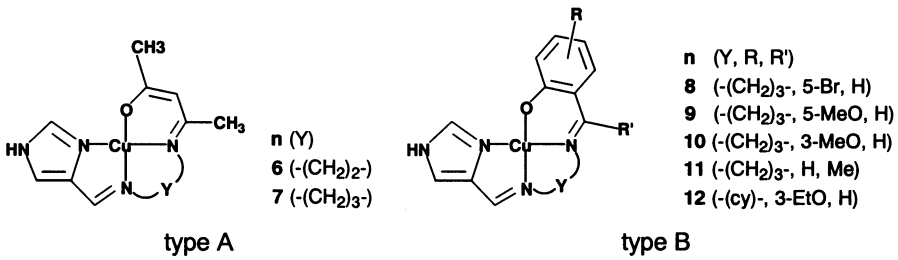
In this article, as a representative example of method **I**, Cu(II) complexes with multidentate Schiff-base ligands containing imidazole groups are discussed. These Cu(II) complexes potentially have both donor and acceptor abilities in the formation of coordination bonding and hydrogen bonding. Under the acidic condition of a pH region lower than the dissociation constant of the imidazole group, the donor ability at the imidazole group is inhibited by the proton and no assembly process occurs. Under an appropriate alkaline condition higher than the dissociation constant, the imidazolate nitrogen atom generated coordinates to a Cu(II) ion of the adjacent unit or hydrogen-bonds to the imidazole group of the adjacent unit to construct the assembly structures. Several factors control the characteristics of the self-assembly process. An appropriate choice of ligand framework may yield a chosen self-assembly structure, due to the independent but additive steric restrictions imposed by the ligand framework and the preferred coordination number and geometry of the Cu(II) ion upon self-assembly. Here we describe the basic conditions for self-complementary molecules.

2. Critical point between ligand-complex and self-complementary molecule: copper(II) complex with a tetradentate ligand containing an imidazole group

Lippard et al. reported that the Cu(II) complex with the tetradentate Schiff-base ligand formed by the 1:1 condensation of 4-formylimidazole and *N*,*N*-dimethyldiethylenetriamine **1**. Compound **1** reacts with pentamethyldiethylenetriaminecopper(II) and with itself in the presence of base to give the imidazolate-bridged species **2** and **3**, respectively [8]. Brewer et al. isolated a mono-deprotonated species **4** [9,10]. This complex **4** functions as a monodentate ligand-complex at the imidazolate nitrogen atom and reacts with tetraphenylporphyrinatoFe(III) to give a linear Cu(II)–Fe(III)–Cu(II) complex **5**, in which a ferromagnetic interaction between low-spin Fe(III) and Cu(II) ions is observed [11].



We have synthesized a series of Cu(II) complexes of the unsymmetrical tetradentate Schiff-base ligands containing an imidazole group per molecule (**6**–**12**) and examined their self-complementary behavior [12–14]. The Cu(II) complexes exhibit potentially the following building-block characteristics, allowing a simple self-assembly process: (1) the complex molecule has potential donor ability at the imidazolate nitrogen atom; (2) the complex molecule has potential acceptor ability at the fifth axial coordination site if it is available; (3) although the complex molecule has potentially both donor and acceptor coordination abilities, the donor ability is concealed by the imidazole proton in acidic conditions lower than pK_d (dissociation constant of imidazole proton) where the complex is thus monomeric; (4) at pH higher than pK_d, the donor ability which triggers the self-assembly process is revealed; (5) since Cu(II) ion prefers either four or five coordination with these tetradentate Schiff-base ligands, only the axial coordination site is available to assemble the species; (6) they can be classified into two types: Type A and Type B complexes, from the deprotonation behavior and by the strength of the equatorial ligand field.



Type A: When the ligand field strength of the equatorial tetradentate ligand is strong enough or there is steric hindrance to axial coordination, the self-assembly reaction does not occur. Such a deprotonated complex would exist as a monomer and behaves as a ligand-complex like Brewer's complex **4**. As shown in Fig. 1, the ligand-complex **6** coordinates to another metal complex exhibiting a vacant and/or substitutable coordination site to give a series of imidazolate-bridged homo- and hetero-metal polynuclear complexes, whose magnetic properties were extensively investigated [15–25].

Type B: When the ligand field strength of the equatorial tetradentate ligand is sufficiently weak and the complex has acceptor ability to receive the imidazolate nitrogen atom of the adjacent unit at the axial coordination site, the self-assembly reaction is motivated by the deprotonation of the imidazole group and the self-assembly compound is formed. For example, the protonated Cu(II) complex **12** exists as a monomer and assumes a square-planar coordination geometry with the N_3O donor atoms of the tetradentate Schiff-base ligand. The deprotonated complex **12′** assumes an imidazolate-bridged one-dimensional (1D) zigzag-chain structure [18]. The self-assembly process from monomer to imidazolate-bridged 1D zigzag chain is schematically shown in Fig. 2.

3. Copper(II) complexes with tridentate ligands containing an imidazole group

Copper(II) complexes with tridentate ligands containing an imidazole group formed by the 1:1 condensation of 2-methyl-4-formylimidazole (or 2-phenyl-4-formylimidazole) and 2-aminoethylpyridine, **13** and **14**, are representative molecules which show pH-dependent reversible interconversion between monomer and oligomer [26]. Compounds **13** and **14** with the chemical formula $[CuCl_2(HL)]$ (HL = *N*-(2-methyl or phenyl-imidazol-4-ylmethylidene)-2-aminoethylpyridine) assume a pentacoordinated geometry with a N_3Cl_2 donor set in the solid state. The Cu–Cl(1) and Cu–Cl(2) distances are 2.293(1) and 2.581(1) Å for **13**, and 2.2679(5) and

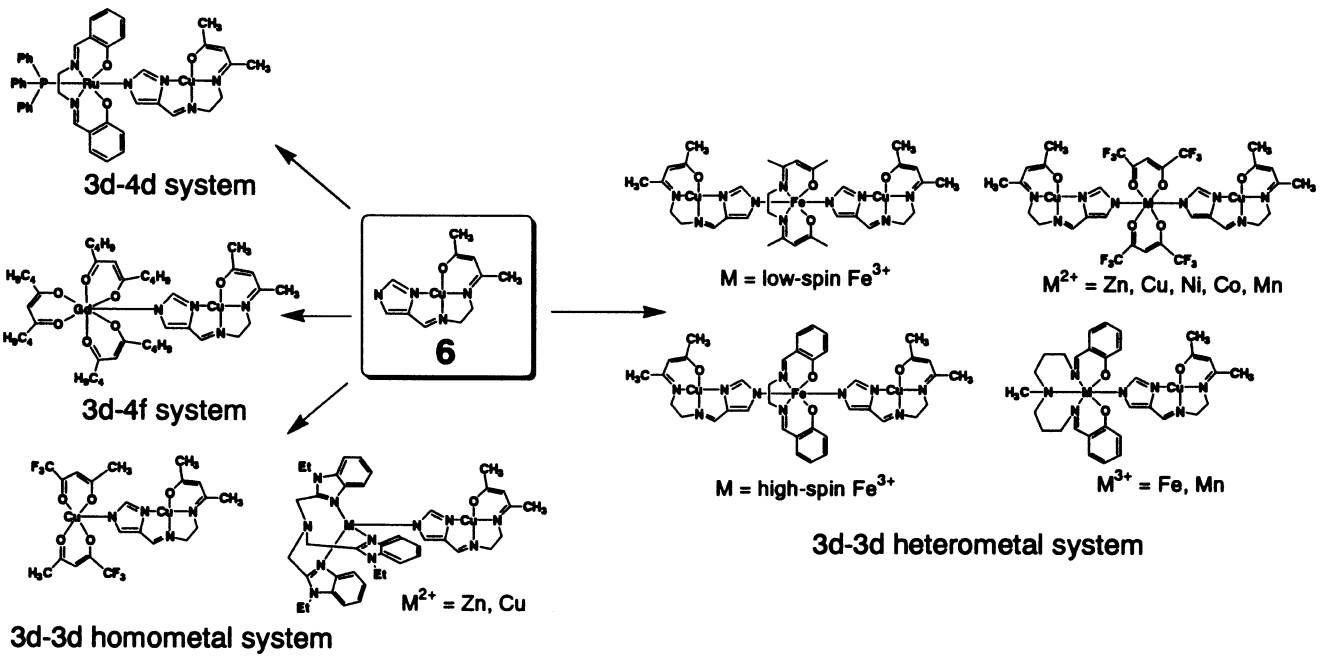


Fig. 1. Reaction scheme of **6** as a *ligand-complex* with the other metal complexes.

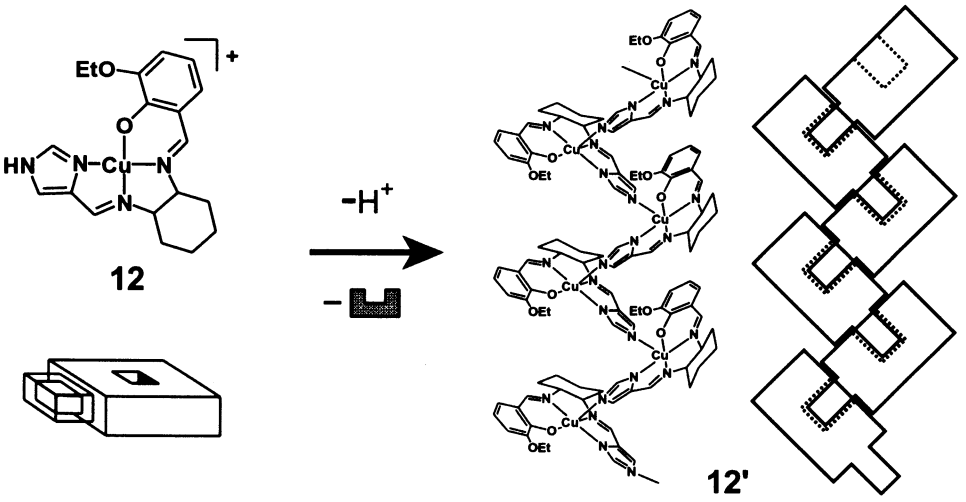


Fig. 2. Schematic representation for the self-assembly reaction of **12**.

2.5184(6) Å for **14**, respectively. In the solid state two Cl^- anions are bound to the Cu(II) ion, but there are no Cu–Cl bonds in aqueous solution where water molecules instead of Cl^- are coordinated to Cu(II). Since the three equatorial coordination sites are occupied by the N_3 donor atoms of the tridentate ligand and the coordinated water molecule is easily substituted, the fourth equatorial site can be used for the assembly reaction. When the protonated complexes **13** and **14** were treated with an equimolar amount of triethylamine or NaOH in aqueous solution, condensation occurred through formation of coordination bonds between the imidazolate nitrogen atoms and Cu(II) ions, yielding the self-assembled cyclic tetranuclear and hexanuclear compounds **13′** and **14′**, in which each Cu(II) ion is coordinated by the three nitrogen donor atoms from the tridentate ligand and the imidazolate nitrogen of the adjacent unit. Fig. 3 shows the assembly reaction from monomer to oligomer, together with the schematic representation by a molecular brick.

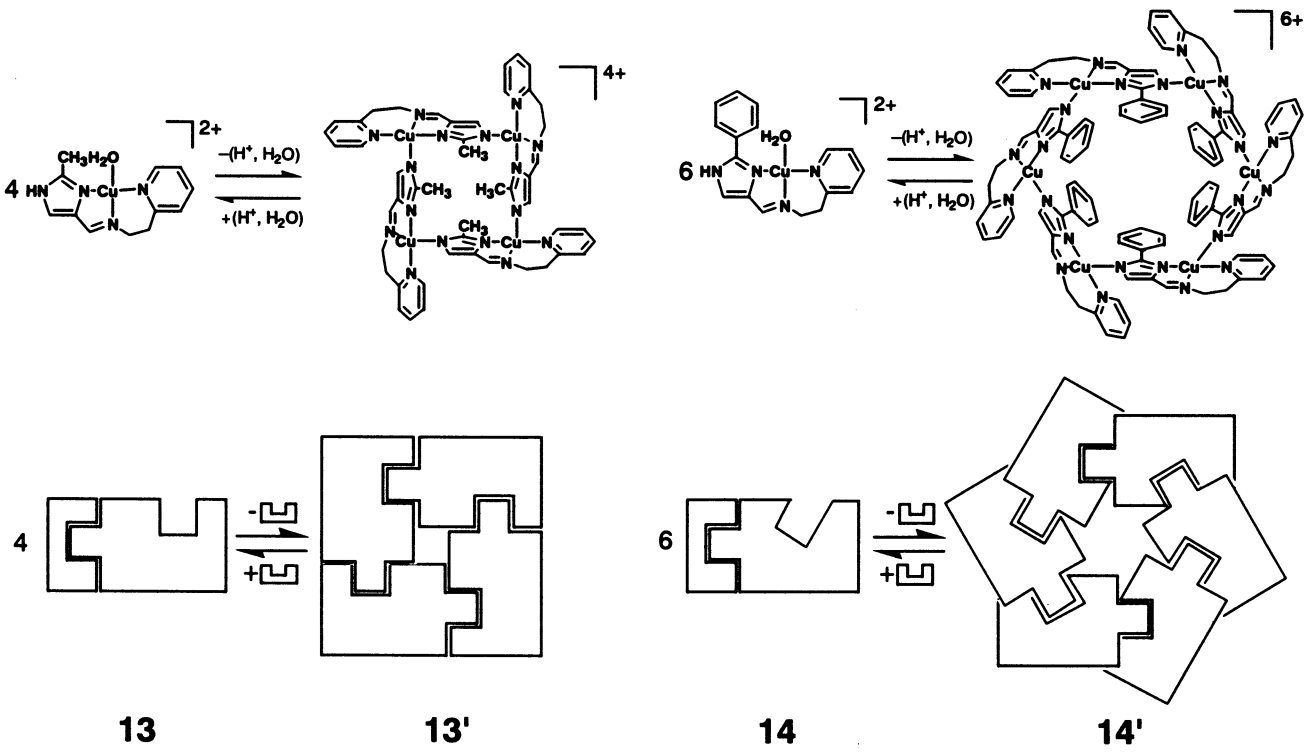


Fig. 3. Schematic representation of the self-assembly reaction for Cu(II) complexes with tridentate ligands containing an imidazole group, **13** and **14**.

There are no relevant differences in the four Cu–N coordination bond distances and imidazolate-bridged Cu–Cu distances between the two oligomeric structures. Among these four Cu–N bonds, the shortest involves the imidazolate nitrogen atom occupying the equatorial coordination site of the adjacent Cu(II) ion. The 2-imidazolate substituents of the adjacent modular units orient themselves in directions opposite to each other to form the imidazolate-bridged cyclic structure. Thus, the 2-imidazole substituents of the next nearest neighboring units face each other. Therefore, concerning the strong coordination bonds resulting from the imidazolate-bridges and the cyclic structure, the steric repulsion between 2-imidazolate substituents facing each other is the factor determining the orientation of adjacent modular units, and consequently the actual structure. A cyclic hexanuclear structure is more favorable for the bulky 2-phenyl substituent than a cyclic tetranuclear structure to avoid steric repulsion, because the Cu–Cu distance of similarly oriented modular units is increased in a cyclic hexanuclear structure. The space-filled drawings (Fig. 4) clearly show that the presence of bulky phenyl substituents does not allow a cyclic tetranuclear structure for steric reasons. The structural parameter determining the nuclearity is the N–Cu–N angle in the $(–Cu–Im)_n$- macrocyclic framework. Thus, the N–Cu–N angle in the **13′** cyclic tetramer is substantially smaller than in the cyclic hexamer **14′**.

The reversible interconversion between the Cu(II) monomer and cyclic oligomer has been confirmed by potentiometric pH titration and pH-dependent electronic spectra. Fig. 5(a) shows the results of forward and reverse titrations carried out in water at 25 °C. Fig. 5(b) shows the pH-dependent electronic spectra for the **13** → **13′** forward conversion. In Fig. 5(a), the degree of proton association n decreases from 1 to 0 for the forward titration and increases from 0 to 1 for the reverse titration. The forward and reverse titration curves are almost the same, indicating that the deprotonation and protonation are reversible. In Fig. 5(b), the spectrum of **13** exhibits a broad band at 682 nm (molar extinction coefficient $\varepsilon = 48\ M^{-1}\ cm^{-1}$) assignable to a

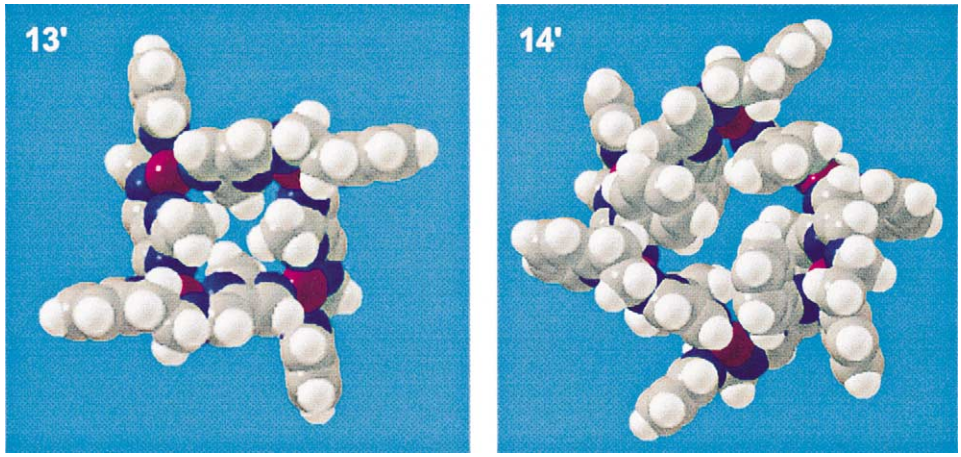


Fig. 4. Space-filling representations of cyclic tetranuclear **13′** (left) and hexanuclear **14′** (right) complex molecules, showing the cavity and the orientation of 2-imidazolate substituents.

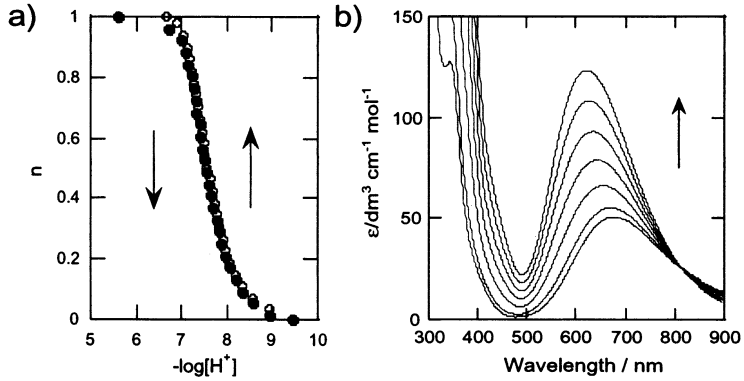
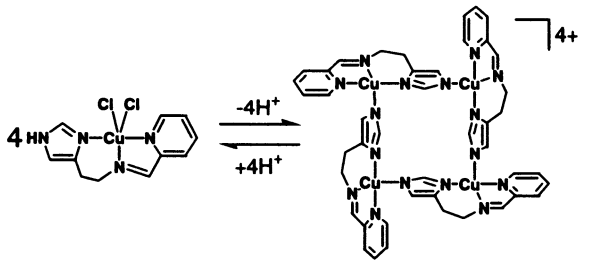


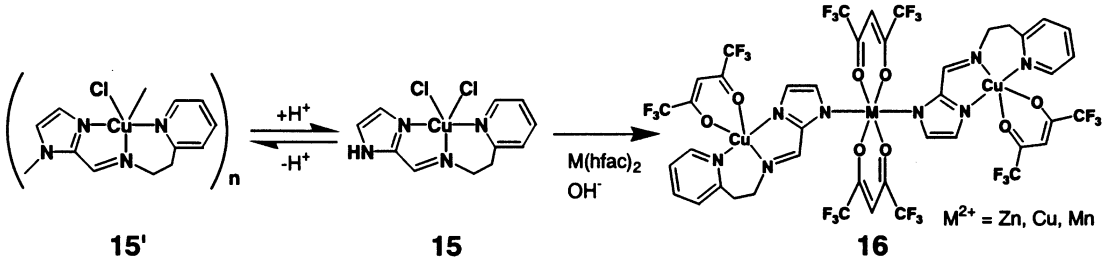
Fig. 5. (a) pH-Dependent potentiometric titration curves (proton association degree n vs. pH) for the forward (black) and reverse (white) titrations of **13**. (b) pH-Dependent electronic spectra of **13** for the forward titration procedure. A spectrum was recorded after each 0.4 ml addition of a 0.1 M NaOH solution, until 1 equiv. of NaOH was added.

d–d transition. On adding a 0.1 M aqueous NaOH solution, this broad band shifts to lower wavelength and the molar extinction coefficient increases. When the amount of NaOH solution added was equimolar, the spectrum generated had a d–d band maximum at 620 nm ($\varepsilon = 127\ M^{-1}\,cm^{-1}$), perfectly consistent with the spectrum of **13′**. This spectral change is characterized by an isosbestic point at 805 nm, typical of an equilibrium between the protonated and deprotonated forms. The spectra for the **13′ → 13** reverse titration exhibit an isosbestic point at the same wavelength and reach the genuine spectrum of **13**. The reversibility is achieved owing to the coordination plasticity of Cu(II).



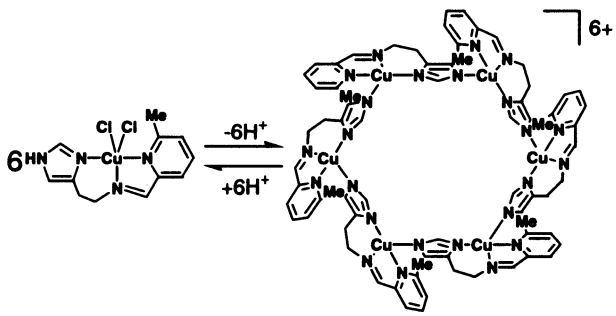
17 **17'**

Coracio et al. synthesized the analogous Cu(II) complex with the tridentate ligand formed by the 1:1 condensation product of 2-formylimidazole and 2-aminoethylpyridine, **15** [27,28]. The Cu(II) complex **15** is different from **13** and **14** in that the tridentate ligand of **15** is derived from 2-formylimidazole, instead of 4-formylimidazole. The self-assembly structure of the deprotonated compound **15′** is an imidazolate-bridged 1D structure, which is quite different from the cyclic structures of **13′** and **14′**. Further, compound **15** reacts with $hfac^-$ under alkaline conditions and the presence of excess $M(hfac)_2$ to give a trinuclear compound **16**.



15' **15** **16**

Nakao et al. synthesized the Cu(II) complex with the tridentate ligand formed by the 1:1 condensation of histamine and 2-pyridinecarboxyaldehyde, **17**. The deprotonation reaction of **17** gave an imidazolate-bridged tetranuclear compound **17′**, whose structure was suggested by the temperature-dependent magnetic susceptibility [29]. We have modified the complex slightly by the use of 6-methyl-2-carboxypyridine, instead of 2-pyridinecarboxyaldehyde, **18**. The deprotonation reaction of **18** gave the imidazolate-bridged cyclic hexamer **18′**. The complex showed pH-dependent reversible interconversion between monomer **18** and cyclic hexamer **18′** [30]. The corresponding Pd(II) complex with the same ligand produced a cyclic tetranuclear structure under deprotonation, showing that the nuclearity depends on the metal ion. The disassembly process also depends on the metal ion, since the Cu(II) complex exhibits a reversible interconversion, whereas the Pd(II) complex did not show a perfect disassembly process [28].



18 **18'**

4. Copper(II) complex with a tetradentate ligand containing two imidazole groups

Copper(II) complexes with *N*,*N′*-bis(2-substituted-imidazol-4-ylmethylidene)-1,4-diaminobutane (2-substituent = H, **19**; Me, **20**) were synthesized [31]. X-ray analyses of **19** and **20** revealed that the molecules assume a butterfly shape bent by a line of N(2)–Cu–N(4) where the angle of N(3)–Cu–N(5) representing the extent of the molecular bend is 150.9(2) and 105.66(8) for **19** and **20**, respectively. Due to the molecular bend, two imidazole moieties (sites A and B) are recognized as two inequivalent groups for the deprotonation process in which the imidazole group of site A is in an equatorial CuN_3 plane, while that of site B is in a CuN_2 plane bent from the equatorial coordination plane. The assembly process is illustrated in Fig. 6. Compound **19** dissociates first a proton from site B to

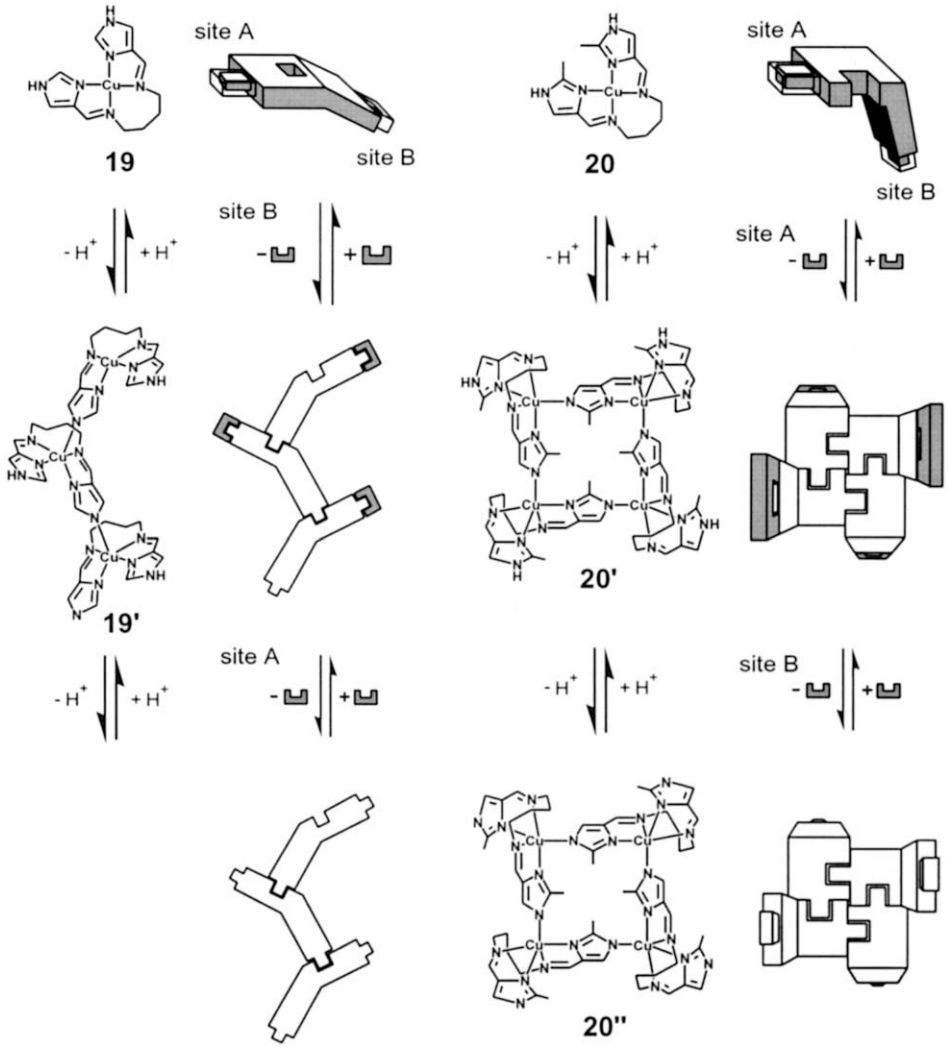


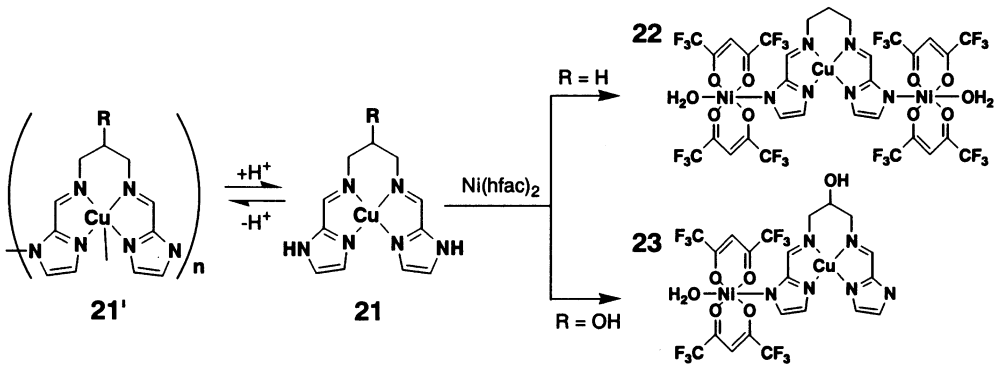
Fig. 6. Schematic representation of the self-assembling reaction for Cu(II) complexes with tetradentate ligands containing two imidazole groups, **19** and **20**.

give an infinite zigzag-chain compound **19′**, while **20** dissociates first a proton from site A to give a cyclic-tetranuclear compound **20′**. Compound **20′** undergoes further deprotonation to give an electrically neutral di-deprotonated complex **20″**. The larger bend found in **20**, **20′**, and **20″** is ascribed to the steric repulsion between two 2-methyl-imidazole moieties. Owing to a nearly perpendicular bend for **20**, the distance of Cu–N(5) = 2.125(2) Å of site B is substantially longer than that of Cu–N(2) = 1.979(2) Å of site A, implying that the imidazole proton at N(1) of site A would dissociate first. The imidazolate nitrogen atom occupies one of the equatorial coordination sites of the adjacent block to give a cyclic tetramer. On the other hand, due to the spread-out shape of **19**, the distance of Cu–N(2) = 1.959(3) Å is rather compatible with that of Cu–N(5) = 1.998(4) Å; in fact, the imidazole proton of site B dissociates first and the imidazolate nitrogen atom, N(6), occupies an apical position of the adjacent molecule to give an infinite chain.

Coracio et al. also reported a copper(II) complex with a tetradentate ligand containing two imidazole groups, **21**, in which 2-formylimidazole is used instead of 2-substituted-4-formylimiazole [32,33]. The deprotonation reaction of **21** gave an imidazolate-bridged 1D compound **21′**, in which the N_4 coordination basal plane is almost planar and the imidazolate nitrogen atom of the adjacent unit occupies the axial position with the Cu–N distance of 2.214(9) Å. Compound **21** also reacts with $Ni(hfac)_2$ to give trinuclear (R = H, **22**) and dinuclear (R = OH, **23**) compounds.

5. Copper(II) complexes with a pentadentate ligand containing two imidazole groups

The protonated copper(II) complexes of the strand-type pentadentate Schiff-base ligands containing two imidazole groups, **24**–**28**, were synthesized in our labo-



ratory [34,35]. When 2 equiv. of triethylamine was added to the 1:1 mixed-solution of the ligand and Cu(II) perchlorate hexahydrate in methanol, the mono-deprotonated complexes **24′**–**28′** were obtained as green crystals. The mono-deprotonated Cu(II) complexes **24′**–**28′** involves protonated (imidazole) and deprotonated (imidazolate) moieties per molecule, in which the Cu–N distance of the protonated moiety is substantially longer than that of the deprotonated moiety, as exemplified by the bond distances of 2.354(6) and 2.000(6) Å for **25′**. Due to the spiral coordination arrangement of the achiral strand-type ligand, the Cu(II) complex becomes a chiral molecule with *C* (clockwise) and *A* (anticlockwise) enantiomers. The mono-deprotonated complex functions as a chiral self-complementary building component and aggregates possibly into heterochiral (…*CACA*…) or homochiral (…*CCCC*… and …*AAAA*…) 1D zigzag chains, due to intermolecular imidazole–imidazolate hydrogen bonds. The chemical structures, the enantiomers, and homochiral and heterochiral 1D structures constructed by the imidazole–imidazolate hydrogen bond are drawn in Fig. 7.

The crystal lattice of **24′**, **25′**, and **26′** yielded either …*CCCC*… or …*AAAA*… isotactic 1D zigzag chains, while the crystal lattices of **27′** and **28′** yielded …*CACA*… syndiotactic 1D zigzag chains. In the former compounds, two adjacent methyl or H groups at the 2-position connected by hydrogen bonds arrayed in the same direction, thus allowing homochiral aggregation of the complex molecules in a 1D chain. On the other hand, in **27′** and **28′**, two adjacent bulky phenyl groups require opposite orientations in order to avoid steric hindrance indicated by the space filling representation (Fig. 8), thus allowing heterochiral aggregation. Enantioselective aggregation with homochirality or heterochirality can thus be controlled with suitable substituents. This assembly reaction involves a process from the synthetically simple achiral ligand and a Cu(II) ion, to the isolated chiral complex, and then to the homochiral and heterochiral 1D zigzag chain. The process is schematically drawn in Fig. 9.

6. Copper(II) complexes with a tripod-type ligand containing three imidazole groups

A copper(II) complex with a hexadentate tripod-type ligand involving three imidazole groups, **29**, induces a chirality of *C* and *A* enantiomers due to the screw coordination arrangement of the tripod-type ligand

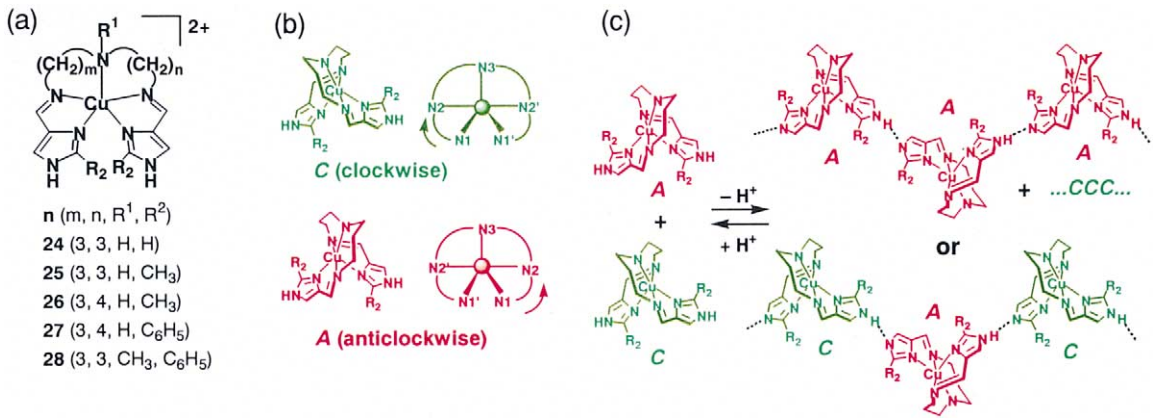


Fig. 7. (a) Molecular structures of copper(II) complexes **24**–**28** with the pentadentate ligands. (b) Structures of *C* (clockwise) and *A* (anticlockwise) enantiomers. (c) Two possible 1D structures of the mono-deprotonated complex formed by hydrogen bonds between two adjacent enantiomers, homochiral (upper) and heterochiral (lower) 1D zigzag chains. Here, *C* and *A* enantiomers represent green and red color, respectively. The same color representation is also used in following figures.

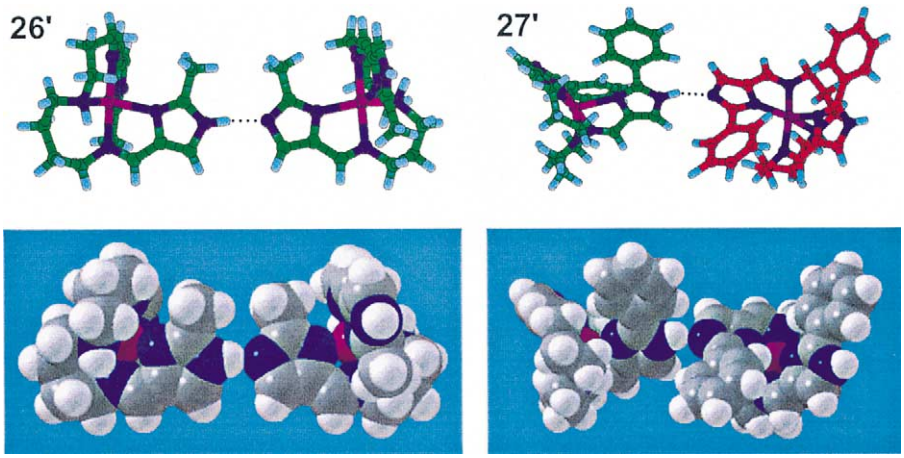


Fig. 8. CHARON drawings and the space filling representations of the two adjacent molecules linked by the hydrogen bond for **26**′ (left) and **27**′ (right).

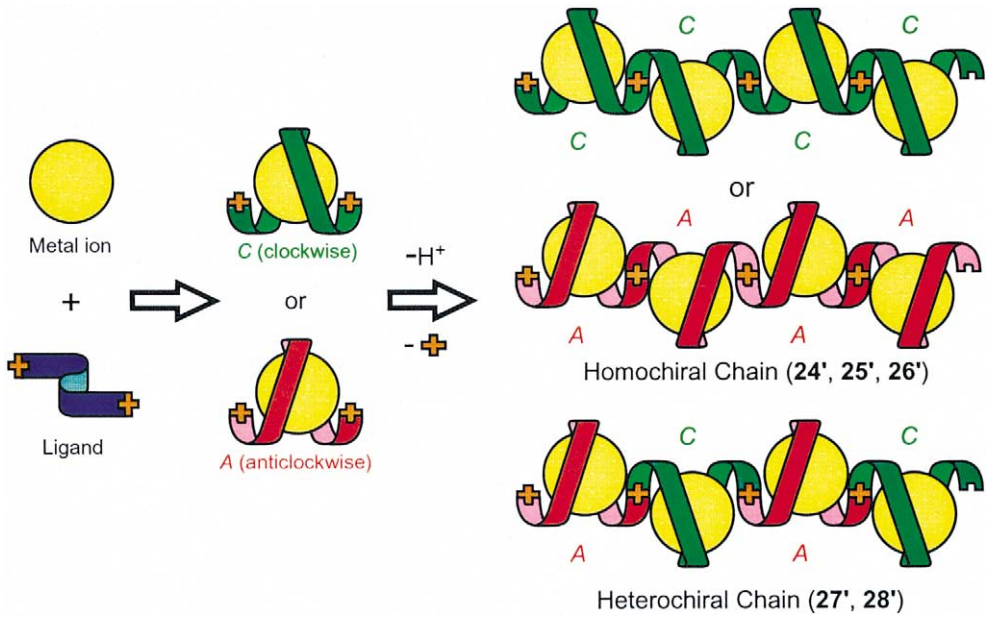


Fig. 9. Schematic representation of the hydrogen bonding self-assembling reaction for Cu(II) complexes with pentadentate ligands containing two imidazole groups, **24**–**28**.

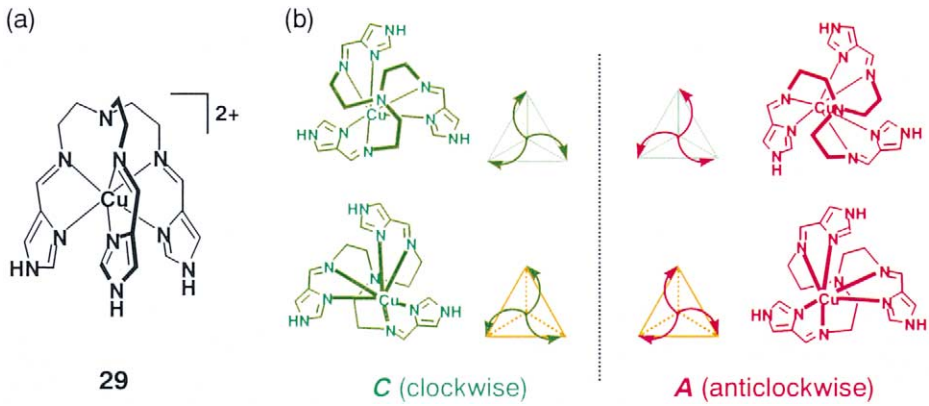


Fig. 10. (a) Molecular structures of $[Cu(H_3L)]^{2+}$. (b) Views looking down and up the molecule along the molecular C_3 axis for *C* and *A* enantiomers and their schematic representations, in which the curve line from the central tertiary amine to the terminal imidazole nitrogen through the imine nitrogen is represented by arrows.

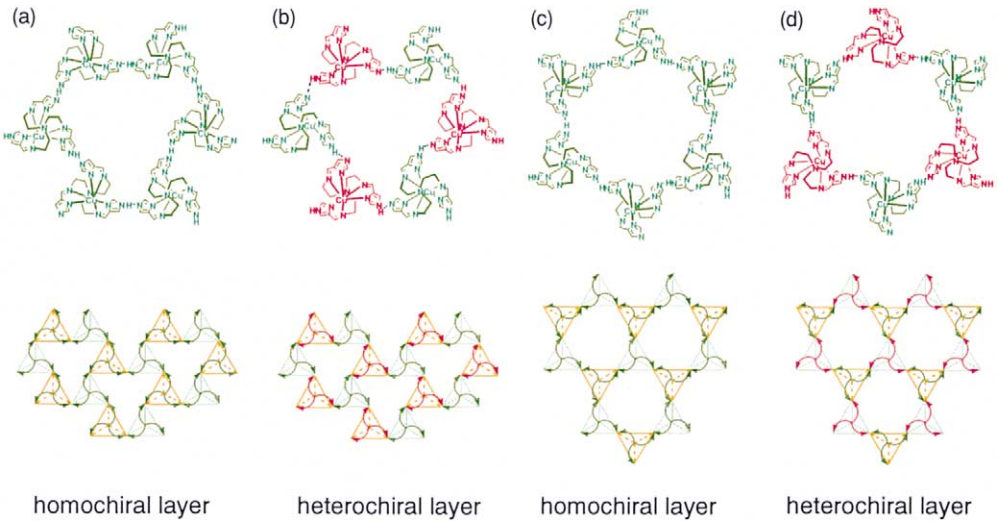


Fig. 11. Two pairs of plausible homochiral and heterochiral assemblies for the hemi-deprotonated complex formed by intermolecular imidazole–imidazolate hydrogen bonds. (a) and (b) show a pair of homochiral and heterochiral structures with trigonal void. (c) and (d) show another pair of homochiral and heterochiral structures with hexagonal void.

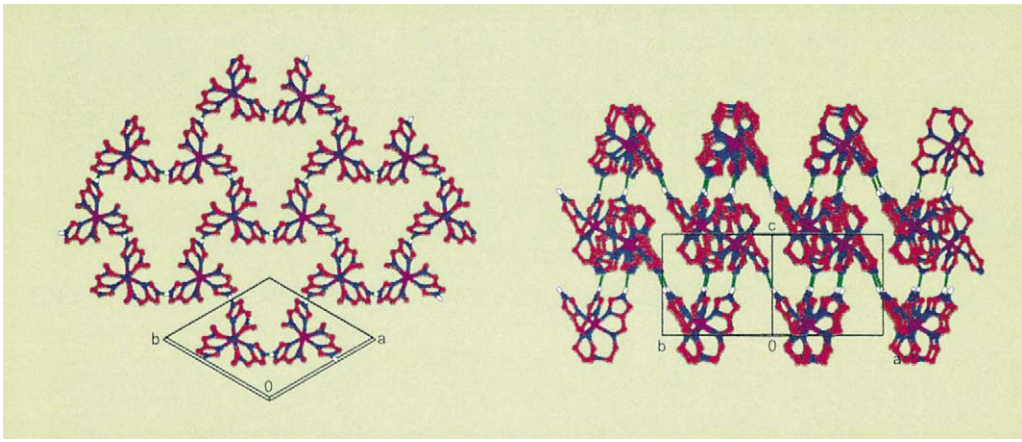


Fig. 12. Chiral two-dimensional structure constructed by hydrogen bonds for **29′** (left) and a side-view showing layer structure (right). Red colored molecule in CHARON drawing represents *A* enantiomer.

around the metal ion as shown in Fig. 10 [36,37]. The hemi-deprotonated species generated in appropriate basic conditions, **29′**, functions as a chiral self-complementary double-faced H-bonding recognition unit and forms a two-dimensional (2D) extended structure due to the intermolecular imidazole–imidazolate hydrogen bond. As shown in Fig. 11, there are four possible structures for a 2D layer (a–d). Among them, only the structure (a) with homochiral aggregation and trigonal void has been so far obtained. The CHARON drawing for the 2D structure of **29′** is shown in Fig. 12. The generated 2D structure is a chiral layer constructed by homochiral aggregation, in which the *C* isomer aggregates only with the *C* isomer and the *A* isomer aggregates only with the *A* isomer to produce a chiral 2D structure. Further, the layers with the same chirality are stacked to form a chiral crystal consisting of homochiral molecules. The X-ray analysis demonstrated that the separation of the two enantiomers spontaneously occurred during the course of the crystallization.

7. Summary

Copper(II) complexes with multidentate Schiff-base ligands containing imidazole groups have been studied. These complexes potentially have both donor and acceptor abilities to function as self-complementary building blocks for the construction of assembly structures or to function as a ligand-complex. In such self-complementary complexes, the monomer is stabilized as a protonated species under acidic conditions, while under appropriate basic conditions the imidazolate nitrogen atom generated coordinates to the metal ion of the

adjacent unit or hydrogen-bonds to the imidazole group of the adjacent unit to give assembly structures in the crystals, depending on the ligand framework and the preferred coordination number and geometry of Cu(II) ion. The deprotonated species of Cu(II) complexes with tetradentate ligands containing an imidazole group function either as self-complementary building blocks to give a imidazolate-bridged zigzag-chain structure or exist as monomers which can be used as a ligand-complex. The deprotonation of Cu(II) complexes with tridentate ligands containing an imidazole group yields cyclic imidazolate-bridged tetranuclear and hexanuclear structures due to the coordination of the imidazolate nitrogen atom to a Cu(II) ion of an adjacent unit. The reversible interconversion between the protonated monomeric and deprotonated oligomeric species were confirmed by pH-dependent potentiometric and electronic spectral titrations in aqueous solution. Cu(II) complexes with tetradentate ligands containing two imidazole groups dissociate a proton to give an infinite zigzag-chain compound or a cyclic-tetranuclear compound, depending on the steric effect. The Cu(II) complex of a pentadentate string-like ligand containing two imidazole groups assumes *C* (clockwise) or *A* (anticlockwise) enantiomers due to the spiral arrangement of the ligand. The mono-deprotonated copper(II) complex containing an imidazole and imidazolate groups per unit functions as a chiral building component for an assembly process due to the intermolecular imidazole–imidazolato hydrogen bond to give homochiral or heterochiral zigzag-chain structures, depending on the steric effect of the substituent. The Cu(II) complex with a hexadentate tripod-type ligand involving three imidazole groups induces a chirality of *C* and *A* enantiomers due to the screw coordination arrangement of the tripod-type ligand. The hemi-deprotonated species or the combination of the fully deprotonated and fully protonated species functions as a chiral self-complementary double-faced H-bonding recognition unit and forms a two-dimensional chiral layer structure.

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