An investigation of a relatively rigid acyclic salamo-type ligand and its square planar Cu(II) complex

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ABSTRACT
A relatively rigid acyclic salamo-type ligand H2L and its square planar Cu(II) complex, [Cu(L)]-CH3OH, were synthesized and characterized by X-ray crystallography, as well as Fourier-transform infrared spectra, UV–Vis spectra, Hirshfeld surface analyses and DFT calculations. The ligand crystallizes in monoclinic space group P 21/c and has pairs of intramolecular hydrogen bonds between the oxime nitrogen atoms and the hydroxyl groups. The Cu(II) complex crystallizes in the monoclinic space group P 21/c. The Cu(II) ion is located in the N2O2 cavity of the fully deprotonated salamo-type ligand (L)2 unit, thus forming a four-coordinate distorted square planar geometry. At the same time, a belt-like, 2-D supramolecular structure is formed by the interaction of intermolecular hydrogen bonds, and the intermolecular interaction force is quantitatively analyzed by Hirshfeld surfaces. Significantly, fluorescence properties of the ligand and its Cu(II) complex were also studied. The coordination ratio of the Cu(II) complex was proved by UV–Vis and fluorescence titration experiments.

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1. Introduction

In recent years, because molecular systems may be applied to the new generation of electronic systems [1, 2], research and use of molecular systems as the prototype of molecular devices has become an important challenge in the scientific community [3–5]. The synthesis and study of such complexes comprising multiple 3d metal ions have generated much interest over the last three decades [6, 7]. The breakthrough in this field is directly related to the development of molecules with new specific characteristics. These molecules can be part of efficient materials (such as molecular switches) or active elements in storage devices or molecular sensors [8–13]. One of the convenient ways to separate these metal ions is to use a ligand that can selectively bind these metal ions [14–19]. The selectivity of this ligand takes advantage of the key differences in size and chemical properties between Cu(II) ions [20–24], which makes the ligand coordinate only with the equatorial plane of these metal ions without coordination position preference [25–28].

Although salen-type ligands have excellent properties, they are extremely unstable in the presence of water and easy to hydrolyze [29–31]. However, salamo-type compounds are derivatives of salen-type ligands. On the other hand, N,O-donor ligands have been used widely for the synthesis of metallic complexes in the past few decades [32–36]. The relatively softer N-donor sites of such ligands bind the soft metal ions and harder O-donor groups select the hard cations. By introducing strongly electronegative O atoms to form –C–N–O– groups, they can form more stable complexes due to their strong coordination abilities [37–41]. This promoted further study of salamo-type ligands and their complexes [42–45]. The complexes have attracted interest because of their unique structures [15] and potential applications in biomedical analysis [37], catalysis [25, 26], molecular/ion recognition [39, 46], and magnetism [6].

Herein, based on our previous works, a relatively rigid salamo-type ligand (H₂L) was designed and synthesized, and its distorted square planar Cu(II) complex has also been synthesized. In addition, the crystal structures, Hirshfeld surfaces analyses and fluorescence properties of H₂L and its Cu(II) complex are also discussed.

2. Experimental

2.1. Materials and methods

8-Hydroxyjulolidine-9-carboxaldehyde was purchased from Mfr: TOKYO Chemical Industry Co. Ltd., Tokyo, Japan; other chemicals were purchased from Tianjin Chemical Reagent plant and used as received. Elemental (C, H, and N) analyses were performed on a GmbH Vario EL version 3.00 automatic elemental analyzer (Elementar, Berlin, Germany). Elemental analysis of Cu(II) was obtained using an IRIS ER/S-WP-1 ICP atomic emission spectrometer (Berlin, Germany). FT-IR spectra were recorded on a VERTEX70 FT-IR spectrophotometer, with samples prepared as KBr (500–4000 cm⁻¹) and CsI (100–500 cm⁻¹) pellets. UV–Vis absorption and fluorescence spectra were recorded on Shimadzu UV-2550 (Shimadzu, Kyoto, Japan) and Hitachi F-7000 (Hitachi, Tokyo, Japan) spectrometers, respectively. ¹H NMR spectra were determined via a Bruker Avance DRX-400/600 spectrometer [21, 25].
2.2. Preparation of (1E,1′E)-O,O′-(ethane-1,2-diyl)bis(1-(8-hydroxy-2,3,6,7-tetrahydro-1H,5H-pyrido[3,2,1-ij]quinolin-9-yl)-8-hydroxy-2,3,6,7-tetrahydro-1H,5H-pyrido[3,2,1-ij]quinoline-9-carbaldehyde oxime) (H₂L)

1,2-Bis(aminooxy)ethane was synthesized following a related previously reported procedure [46, 47]. 8-Hydroxyjulolidine-9-carboxaldehyde (434.5 mg, 2.0 mmol) was dissolved in 40 mL of EtOH to get a clear solution. To this was added an ethanol solution (20 mL) of 1,2-bis(aminooxy)ethane (92.1 mg, 1.0 mmol) dropwise; the reaction mixture was heated and stirred for about 4 h. On cooling to room temperature, excess ethanol was removed by rotary evaporation to obtain the light red solid. This solid was re-dissolved in chloroform and purified by recrystallization in n-hexane. Yield: 371.9 mg (75.8%). M.p. 161–162°C. Anal. Calcd. for C₂₈H₃₄N₄O₄ (%): C, 68.55; H, 6.99; N, 11.42. Found: C, 68.76; H, 6.81; N, 11.25. IR (KBr; cm⁻¹): 3448 (m), 2932 (w), 2848 (w), 1627 (s), 1509 (s), 1315 (s), 1221 (s), 1176 (s), 1054 (w), 948 (w), 898 (m), 670 (m), 431 (w).

H₂L (2.5 mg, 0.005 mmol) was dissolved in a CHCl₃/MeOH (v/v, 2:4) mixed solution (6 mL) and after stirring for 10 min, the solution was filtered, the filtrate was moved to a vial which was sealed with foil, and after standing at room temperature for about 7 d, colorless transparent block-shaped crystals suitable for X-ray diffraction analysis were obtained.

2.3. Synthesis of (1E,1′E)-O,O′-(ethane-1,2-diyl)bis(1-(8-hydroxy-2,3,6,7-tetrahydro-1H,5H-pyrido[3,2,1-ij]quinolin-9-yl)-8-hydroxy-2,3,6,7-tetrahydro-1H,5H-pyrido[3,2,1-ij]quinoline-9-carbaldehyde oxime) [di-phenoxy-copper(II)] (Cu(II) complex)

H₂L (2.5 mg, 0.005 mmol) was added into 2.0 mL of CHCl₃ to form a clear red solution. The resulting solution was stirred for 5 minutes more, and then Cu(OAc)₂·4H₂O (2.0 mg, 0.01 mmol) dissolved in methanol (6.0 mL) was added and stirred for another 10 min. The mixed solution was filtered. After 2 weeks, dark brown crystals suitable for X-ray crystallography measurement were isolated from the slow evaporation of the solvent. Yield: 0.99 mg (34.0%). Anal. Calcd. for [Cu(L)]·CH₃OH (C₂₉H₃₆CuN₄O₅) (%): C, 59.63; H, 6.21; Cu, 10.88; N, 9.59. Found: C, 59.52; H, 6.35; Cu, 10.95; N, 9.64. IR (KBr; cm⁻¹): 3426 (m), 2932 (w), 2843 (w), 1611 (s), 1514 (s), 1307 (s), 1243 (s), 1176 (s), 1054 (w), 948 (w), 898 (m), 670 (m), 469 (w), 462 (w). λₘₐₓ (nm) (εₘₐₓ, L·mol⁻¹·cm⁻¹): 233 (4.89 × 10⁴); 263 (2.46 × 10⁴); 348 (1.18 × 10⁵).

Although a reaction ratio of 1:1 (ligand:copper(II) salt) was also used during the growth of single crystals, no single crystal with a coordination ratio of 1:1 (ligand:copper(II)) was obtained.

2.4. X-ray structure determinations of the Cu(II) complex

The single-crystal X-ray diffraction data were collected at 100 K for the ligand and its Cu(II) complex on a Bruker D8 Venture diffractometer equipped with a graphite-monochromated Mo-Kα radiation source (λ = 0.71073 Å). The coordinates of the non-hydrogen atoms were refined anisotropically, whereas hydrogen atoms bonded to carbon
atoms were included in the calculation isotropically but not refined. Data collection was performed with the APEX3 software package and the SAINT, SMART, and XPREP software packages have been used for the index, integration, and space-group determination of each frame [48]. Absorption corrections were applied using the multiscan program SADABS [49]. The crystal structure of H2L was solved by the direct method and refined by the full-matrix least-squares method using the SHELXT-2014/5 [50] and SHELXL-2018/3 programs [51] implemented in the OLEX2 program [52]. The crystal structure of the Cu(II) complex was solved by the direct method and refined by the full-matrix least-squares method used the SHELXT and SHELXL-2018/3 programs implemented in OLEX2 program. H3AA, H3AB, H3BC, and H3BD atoms are disordered located on the C3 atom. The C3 and C5 atoms of the Cu(II) complex are not disordered, but the hydrogen atoms above them are disordered unequally in two different positions. The probability that H3AA and H3AB atoms are occupied is 0.857(4), and the same H3BC and H3BD atoms are occupied by 0.143(4). H4A and H4B atoms are disordered located on C4 atom, the probability of H4A and H4B atoms being occupied is 0.857(4). H4AA and H4AB atoms are disordered located on C4A atom, the probability of H4AA and H4AB atoms being occupied is 0.143(4). The C4 atom and hydrogen atoms of the Cu(II) complex are disordered unequally over two sites (C4, H4A and H4B, 0.857(4); C4A, H4AA, and H4AB, 0.143(4)), which are allowed for during refinement [53]. The crystal data and refinement parameters for the ligand and its Cu(II) complex are summarized in Table 1.

| Crystal data and refinement parameters for H2L and its Cu(II) complex. |
|---------------------------------------------------------------|
| **H2L** | **Cu(II) complex** |
| Empirical formula | C28H34N4O4 | C29H36CuN4O5 |
| Molecular weight | 490.59 | 584.16 |
| Crystal size (mm) | 0.140 × 0.110 × 0.090 | 0.25 × 0.22 × 0.21 |
| Crystal description | Block-shaped | Block-shaped |
| Crystal system | Monoclinic | Monoclinic |
| Space group | P 21/c | P 21/c |
| Unit cell dimensions | | |
| a (Å) | 5.1584(3) | 10.1609(3) |
| b (Å) | 28.0288(15) | 17.1420(6) |
| c (Å) | 8.8434(4) | 16.1061(5) |
| α (°) | 90 | 90 |
| β (°) | 106.275(2) | 103.9410(10) |
| γ (°) | 90 | 90 |
| V (Å³) | 1227.37(11) | 2722.70(15) |
| Z | 2 | 2 |
| Dcalc (g cm⁻³) | 1.327 | 1.425 |
| µ (mm⁻¹) | 0.090 | 0.849 |
| F(000) | 524.0 | 1228.0 |
| θ Range for data collection (°) | 2.507–26.789 | 2.363–26.75 |
| Index ranges | | |
| h | −6 ≤ h ≤ 6, | −12 ≤ h ≤ 11, |
| k | −35 ≤ k ≤ 35, | −21 ≤ k ≤ 21, |
| l | −11 ≤ l ≤ 11 | −20 ≤ l ≤ 20 |
| Reflections collected | 15271 | 50836 |
| Completeness to (%) (θ) | 99.7 | 99.6 |
| Data/restraints/parameters | 2628/1/167 | 5779/0/358 |
| Final R₁, wR₂ indices | 0.0547, 0.1185 | 0.0282, 0.0795 |
| R₁, wR₂ indices (all data) | 0.0833, 0.1345 | 0.0314, 0.0816 |
| Largest diff. peak and hole (e Å⁻³) | 0.42, −0.22 | 0.80, −0.33 |
3. Results and discussion

The reaction steps involved in the synthesis of the salamo-type ligand H₂L are shown in Scheme 1. H₂L was prepared by 1:2 condensation of 1,2-bis(aminooxy)ethane and 8-hydroxyjulolidine-9-carboxaldehyde in ethanol.

3.1. IR spectral analyses

The IR spectra of H₂L and its Cu(II) complex are highlighted in Figure S1. In the IR spectra of H₂L and its Cu(II) complex, one broad strong peak for the O–H stretching vibration appeared at 3448 cm⁻¹, while the corresponding absorption band of the Cu(II) complex disappeared, indicating that the phenolic hydroxyl groups of H₂L completely deprotonated and coordinated with the Cu(II) ion. A strong stretching vibration of the Cu(II) complex was found at 3426 cm⁻¹, which is assigned to the crystallized methanol molecule [38]. For the ligand, one strong and sharp peak for the C= N stretching vibration appeared at 1627 cm⁻¹. This band is shifted by 16 cm⁻¹ to lower wavenumber in the Cu(II) complex, indicating that the Cu(II) ion has bonded with N atoms of L [38]. The Ar–O stretching vibration frequency of the Cu(II) complex was found at 1243 cm⁻¹ and moved to lower frequency by 22 cm⁻¹ compared to H₂L, which may be evidence of the formation of a Cu–O bond between the Cu(II) ion and phenoxy oxygen atoms [54, 55].

In addition, the far-infrared spectrum of the Cu(II) complex was measured from 500 to 100 cm⁻¹. For the Cu(II) complex, the absorption band at 469 cm⁻¹ further confirms the presence of stretching vibration of the Cu–N bonds, while a broad band at 462 cm⁻¹ can be regarded as the Cu–O vibrations.

3.2. UV spectral analyses

The electronic absorption spectra recorded for the free ligand and its Cu(II) complex in ethanol are depicted in Figure 1. Three UV absorption bands of free H₂L were observed at ca. 233, 263, and 348 nm, which have changed in the UV absorption spectrum of the Cu(II) complex [56]. The former peaks at 233 and 263 nm can be attributed to the π−π* transitions of the benzene rings, while the latter peak at 348 nm can be assigned to intra-ligand π−π* transition of the oxime groups [57]. During gradual addition of Cu²⁺, the absorption intensity of the bands of the free ligand at 233, 263, and 348 nm decreased, while new absorption bands centered at ca. 241, 280, and 366 nm appeared with increasing intensities, which are attributable to formation of the desired...
Cu(II) complex. Well-anchored isosbestic points appeared at ca. 265, 299, and 355 nm for the Cu(II) complex, respectively (Figure 1(b)), and indicated the existence of an equilibrium between the corresponding ligand and the Cu(II) complex in solution [58]. The titration curve showed the formation of a 1:1 mononuclear Cu(II) complex and is shown in Figure 1(b).

### 3.3. Crystal structure descriptions

The crystal structures of H₂L and its Cu(II) complex were demonstrated by X-ray crystallography. Selected bond lengths and angles are listed in Table 2.

#### 3.3.1. Crystal structure of H₂L

The molecular structure of H₂L including one pair of obvious intramolecular hydrogen bonds (O₂–H₂⋯N₁ and O₂#1–H₂#1⋯N₁#1) [59] in the ligand H₂L molecule are shown in Figure 2. H₂L crystallizes in the monoclinic space group P 2₁/c. Further analysis of the crystal structure showed that H₂L has a pair of intramolecular hydrogen bonds between the oxime nitrogen atoms and the hydroxyl groups. There was no observed intermolecular bonding in the packing of the ligand. The almost complete coplanarity

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**Table 2.** Selected bond lengths (Å) and angles (°) for H₂L and its Cu(II) complex.

| Bond       | H₂L Lengths | Cu(II) Complex Lengths |
|------------|--------------|-------------------------|
| O₁–N₁     | 1.420(2)     | Cu₁–O₁ 1.8949(12)       |
| N₁–C₂     | 1.282(3)     | Cu₁–N₂ 1.9722(14)       |
| N₂–C₁₂    | 1.446(3)     | Cu₁–N₃ 1.9722(14)       |
| O₁#1–C₁#1 | 1.430(24)    | Cu₁–O₄ 1.9119(14)       |
| O₂#1–C₄#1 | 1.363(21)    |                       |

Symmetry transformations used to generate equivalent atoms: #₁ = −1 − x, 1 − y, and − z.
of the unsaturated fragments of H$_2$L is indicative of its highly conjugated $\pi$-electronic system conducive to charge transfer transition across the molecule. It can be seen from the crystal structure of the ligand that the configuration of the ligand is Z-shaped.

3.3.2. Crystal structure of the Cu(II) complex

The structure of the Cu(II) complex is shown in Figure 3. The Cu(II) complex crystallizes in the monoclinic crystal system, space group $P 2_1/c$, and includes one Cu(II) ion and one deprotonated ligand (L)$_2$ unit. The Cu(II) ion is four-coordinate [59], its coordination environment is saturated by the two phenol oxygen atoms (O1 and O4) and the two oxime nitrogen atoms (N1 and N4) from the N$_2$O$_2$-donor cavity of the ligand (L)$_2$ unit (Table 2). The dihedral angle of N2–Cu1–O1 and N3–Cu1–O4 in the Cu(II) complex is 27.41(3)$^\circ$, indicating that the Cu(II) ion possesses near square planar geometry which is distorted tetrahedrally by 27.41(3)$^\circ$. Similar distortion was also observed for methoxy-substituted derivative Cu(3-MeOsalamo) which showed more distortion (41.05(17)$^\circ$) [60].

By studying the intermolecular hydrogen bonds between the Cu(II) complex molecules, only three types of hydrogen bonds between the Cu(II) complex molecules were found, C16–H16⋯O5, O5 – H5⋯O1 and O5 – H5⋯O4 (Figure 4(a)) [61]. In the crystal packing, the chains are stacked with weak intermolecular C–H⋯π interactions [62] between adjacent benzene rings and atoms of the ligand (L)$_2$ units (Figure 4(b)). The Cu(II) complex displays a belt-like, 2-D supramolecular structure (Figure 5) [63].
3.4. Hirshfeld surfaces analyses

To gain deeper insight into the supramolecular contacts in the Cu(II) complex, we undertook Hirshfeld surface analysis using CrystalExplorer17 [64]. The Hirshfeld surfaces of the Cu(II) complex are shown in Figure 6. The red spots represent the strongest interactions in the structures and are similar across the family, including C–H⋯O, C–H⋯π, and π–π interactions [65]. The shape index is a qualitative measure of the shape of the surface. It allows the visualization of complementary surfaces; red areas
supplement blue areas of a nearby surface. The shape index in the Cu(II) complex and its co-crystals present the expected equal triangles for the interaction between the aromatic rings. A surface with low curvedness designates a flat region and may be indicative of \( \pi-\pi \) stacking in the crystal. On the other hand, Hirshfeld surfaces with high curvedness are highlighted as dark-blue edges, all of which are indicative of an absence of \( \pi-\pi \) stacking.

The 2-D fingerprint plots [66] allow the relative contributions of these interactions to be determined with the results shown in Figure 7. We begin with the Cu(II) complex, where the fingerprint plots revealed that there are strong C–H⋯O interactions, as indicated by the sharp peaks in the plot. In all cases, we find that the H⋯O and O⋯H interactions contribute significantly to the supramolecular makeup of the compounds, being 12.9% in the Cu(II) complex. The forcep-like tips in the fingerprint area represent C–H–H⋯C contacts, in which the proportion of total C–H to Hirshfeld surface is 15.3%. The proportion of C⋯C interactions is 1.5%.

3.5. Fluorescence properties

Figure 8 displays the fluorescence response of the Cu(II) complex in ethanol solution (1.0 \( \times \) 10\(^{-5} \)mol/L). Figure 8(a) shows that at excitation wavelength of 321 nm, the Cu(II) complex has an emission peak at 395 nm. The Cu(II) complex has a bathochromic-shift of 5 nm compared with that of \( \text{H}_2\text{L} \). The influence of Cu\(^{2+} \) on the emission of \( \text{H}_2\text{L} \) was investigated in ethanol, and \( \text{H}_2\text{L} \) showed significant decreasing in fluorescence intensity. The ligand gives an emission peak, at excitation wavelength of 312 nm, at approximately 390 nm, which is mainly caused by the \( \pi-\pi^* \) electronic
transition within the ligand molecule [67]. In the fluorescence titration experiment (Figure 8(b)), incremental addition of Cu$^{2+}$ ions to a solution of H$_2$L, in aliquots of 2 μL for Cu$^{2+}$, was followed by fluorescence spectral measurement. The fluorescence intensity of H$_2$L decreased with increasing concentration of the Cu$^{2+}$ ion, but the position of the emission band had a red-shift. No further changes in intensity were observed in excess addition of 20 μL for Cu$^{2+}$. In this case, fluorescence decrease occurred via electron donor-acceptor or weak charge transfer interactions, which leads to depopulation of the excited states of the fluorophore. The results of fluorescence titration spectra showed that coordination ratio of the ligand to the transition Cu$^{2+}$ ions is 1:1.

3.6. DFT theoretical calculation

Density functional theory (DFT) is a quantum mechanical method to study the electronic structure of multi-electron system, which is widely used in chemistry and physics. To better understand the coordination behavior between Cu(II) ion and H$_2$L in the Cu(II) complex, and further understand the geometric and electronic structure of the ligand and its Cu(II) complex, a preliminary DFT calculation was carried out using Gauss 09 package B3LYP method and the 6–31G (d,p) basis set (Figure 9).

A large HOMO–LUMO gap (ΔE = E$_{\text{LUMO}}$ − E$_{\text{HOMO}}$) can be associated with high kinetic stability and low chemical reactivity [15]. The energy gap ΔE between HOMO and LUMO in H$_2$L was 4.05 eV. In the Cu(II) complex, due to the existence of single electrons in the HOMO and LUMO orbitals of the molecule, they are split into $\alpha$ and $\beta$ orbitals. The energy differences between $\alpha$ and $\beta$ orbits are 1.05 and 0.99 eV, respectively. According to these results, the electronic transitions of H$_2$L and the Cu(II) complex originating from the promotion of electrons from HOMO to LUMO are $\pi$–$\pi^*$ electronic transitions.

4. Conclusion

Single crystals of H$_2$L and its Cu(II) complex have been grown up successfully, and characterized by single-crystal X-ray diffraction, FT–IR, UV spectroscopy, Hirshfeld
surface analyses, and DFT calculations. In the Cu(II) complex [Cu(L)]-CH3OH, one Cu(II) ion is surrounded by the completely deprotonated salamo-type ligand (L)2− unit. The X-ray crystal structure showed that the Cu(II) complex is a four-coordinate distorted square planar configuration. Intermolecular interactions such as hydrogen bonds are formed between neighboring molecules in the Cu(II) complex, resulting in an open 2-D supramolecular network structure. The Hirshfeld surface analysis and fluorescence properties of the Cu(II) complex were also studied. According to Hirshfeld surfaces and 2-D fingerprint plot analyses, the intermolecular interactions of the Cu(II) complex were analyzed, so that the Cu(II) complex is more stable. The relative stability of the Cu(II) complex and the ligand was compared by DFT theoretical calculation. It can be determined by fluorescence titration and UV titration equipment that the coordination ratio of H2L to the Cu(II) ion in the Cu(II) complex is 1:1.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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