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A New 2-((Z)-Thiosemicarbazidomethyl)-Quinolin-8-YL Acetate Ligand and its Cu(II) Complex: Syntheses, Structures, and Characterizations

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A NEW 2-((Z)-THIOSEMICARBAZIDOMETHYL)-QUINOLIN-8-YL ACETATE LIGAND AND ITS Cu(II) COMPLEX: SYNTHESES, STRUCTURES, AND CHARACTERIZATIONS

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GRAPHICAL ABSTRACT



Abstract A new ligand, 2-((Z)-thiosemicarbazidomethyl)-quinolin-8-yl acetate ($C_{13}H_{12}$ N_4O_2S) (L) and its Cu(II) coordination complex, [Cu(L₁)] [HL₁ = (Z)-1-((8-hydroxyquinolin-2-yl)methylene)thiosemicarbazide], have been synthesized, and characterized by IR, elemental analysis, ¹H NMR, MS, and X-ray single-crystal diffraction. L shows a stable three-dimensional supramolecular structure through hydrogen bonds and π - π stacking interactions. [Cu(L₁)] shows a three-dimensional network structure formed only by hydrogen bonds. In addition, the fluorescence spectra of L were measured.

Keywords 8-Hydroxyquinoline derivatives; crystal structure; fluorescence property; sulfur

INTRODUCTION

The rational design and synthesis of light-emitting materials have remained an important research focus in recent years.^{1–3} As with other typical light-emitting materials, luminescent coordination compounds are of great interest due to their good performance in sensor technologies, electro luminescent devices and full-color flat panel displays.^{4,5} However, there is still a challenge in predicting their structures. Generally, there are various factors affecting the final structures, including the organic ligands, central metal ions, and solvent molecules. The selection of the ligand plays a key role because deliberate modifications on the organic ligands can dramatically influence the ultimate structural topologies of the coordination solids. 8-hydroxyquinoline derivatives, with high fluorescence quantum yields, have been used as an object doped in the body of light-emitting materials.^{6–8} Until

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now, the biological activity of sulfur-containing quinoline derivative Schiff base has been researched in related fields,^{9,10} however, the research on their optical properties, such as fluorescence, are few. The variety of possible Schiff-base metal complexes with a wide choice of ligands, and coordination modes, has prompted us to undertake research in this area.

In this paper, we report the synthesis and characterization of a new sulfur-containing Schiff-base ligand 2-((Z)-thiosemicarbazidomethyl) quinolin-8-yl acetate (Scheme 1) and its new copper (II) coordination complex. In addition, the crystal structures of them and the luminescent property of **L** are presented.



Scheme 1

RESULTS AND DISCUSSION

The synthetic route of **L** and coordination complex $[Cu(L_1)]$ is outlined in Scheme 1. All known compounds $(2,^{11,12} 3^{11,13})$ were characterized and the data compared to the literature.

Spectroscopic Properties

The IR spectrum of L shows two bands at 3406 and 3039 cm⁻¹, which are attributed to the two stretching frequencies v(N-H). A strong band at 1761 cm⁻¹ is assigned to the v(C=O) vibration. A strong band at 1591 cm⁻¹ is ascribed to the v(C=N) vibration. The two bands at 1190 and 1163 cm⁻¹ are attributed to the stretching frequencies v(C-O-C). A weak band at 1532 cm⁻¹ is ascribed to the v(C-S-C) vibration. A weak band at 846 cm⁻¹ is ascribed to the v(C=S) vibration. The IR spectra of L are consistent with the structure of L.

The IR spectrum of $[Cu(L_1)]$ has been employed to distinguish the coordination modes of copper (II) ion. In the IR spectrum, v(N-H) absorption bands appear at 3422 cm⁻¹. The v(C=N) absorption band appears at 1633 cm⁻¹, which has a 42 cm⁻¹ red shift compared with 1591 cm⁻¹ in free **L**, indicating coordination of the C=N with copper (II). The IR spectra of $[Cu(L_1)]$ are consistent with the structure of $[Cu(L_1)]$.

Crystal Structure

The X-ray crystal structure of **L** with atomic labeling scheme is shown in Figure 1 (left). The crystal packing of molecules connected by π - π interactions is shown in Figure S1 (Supplemental materials). As shown in Figure 1, the asymmetric unit contains four molecules. Several interesting observations can be found about the structure. First, **L** is composed of acetoxy group, thiosemicarbazide skeleton and quinoline skeleton. The length of C=N is 1.294(2) Å, C=O bond is 1.204(2) Å, C=S bond is 1.6822(17) Å. All the quinoline rings in the molecules show the usual features.



Figure 1 3D ORTEP representation of **L** connected by hydrogen bonds with 30% probability thermal ellipsoids $(A \ 1 - x, 2 - y, 1 - z; B \ 2 - x, 0.5 + y, 1.5 - z; C \ x, 2.5 - y, -0.5 + z)$.

The intramolecular hydrogen bonds such as $N(3)-H(3)\cdots N(1)$ hydrogen bond, is 2.018 Å, 133.1° in the molecules structure of L, which makes C11, C12, N2, N3, H3, N1 an irregular six-membered ring (Figure 1), and makes the six-membered ring and the adjacent pyridyl ring almost coplanar. So is the intramolecular hydrogen bond $N(4)-H(4A)\cdots N(2)$ bond. It is 2.238 Å, 105.4°, which makes N3, C13, N4, H4A, N2 an irregular five-membered ring (Figure 1). The interaction of the two intramolecular hydrogen bonds, makes molecules form a stable cis-configuration, and H3 of N3 and H4A of N4 in ¹H NMR shift to low-field.

From Figure 1, the intermolecular hydrogen bond C(9C)—H(9C)···O(1B), makes the two molecules connect dislocation in parallel. C(10)—H(10)···S(1B), contacts the two molecules vertically. N(4)—H(4B)···S(1A) connects the adjacent molecules in parallel. The single molecules between each other are connected by hydrogen bonds. The hydrogen bonds show three different environments (Figure 1). In the whole structure, there is a weak face-to-face π - π interaction between the phenyl ring plane with the relative pyridyl ring plane, so the molecules are packed into layers, indicated by the interplanar distance of 3.740 Å. Furthermore, the final three-dimensional networks are formed by intramolecular and intermolecular hydrogen bonds interaction, which further stabilize the solid three-dimensional supramolecular structure (Figure S1, Table S1, in Supplemental materials).

The ORTEP representation of $[Cu(L_1)]$ of the complex $[Cu(L_1)]$ is shown in Figure 2. Three-dimensional network structure of the complex $[Cu(L_1)]$ is shown in Figure S2 (Supplemental materials). Selected bond lengths and angles are presented in Table 2. From Figure 2 and Table 2 it can be seen that the copper (II) atom is bonded to two N donors, one O donor, and one S donor of one ligand, displaying a distorted tetrahedral geometry,



Figure 2 ORTEP representation of $[Cu(L_1)]$ showing the molecular structure with 30% probability thermal ellipsoids.

and a methanol molecule is uncoordinated to copper (II) atom in the complex. The distance of the copper(II) ion and the bridging sulfur atom is 2.439(3) Å, which is longer than the bond lengths of Cu(1)–N(1), Cu(1)–N(3) and Cu(1)–O(1) [Cu(1)–N(1) = 1.943(5) Å, Cu(1)–N(3) = 1.889(5) Å, Cu(1)–O(1) = 1.956(4) Å]. The bite angles formed by the donor-metal-donor at the metal center [N(3)–Cu(1)–N(1), 89.19(18)°; N(3)–Cu(1)–S(1), 156.63(14)°; N(1)–Cu(1)–O(1), 85.15(17)°; O(1)–Cu(1)–S(1), 114.85(11)°], which is larger or smaller than the right angle (90°) of a normal tetrahedron, indicating a rather distored tetrahedral coordination geometry around copper (II). As expected the six-membered ring (N1–C8–C10–N2–N3–Cu1) is coplanar with the largest deviation from the plane being 0.0207 Å, inclined at 2.2° to the quinolinone fused ring. The five-membered ring (C1–C9–N1–Cu1–O1) and four-membered ring (C11–N3–Cu1–S1) are similar to the six-membered ring above. In addition, the complex was linked to the methanol molecules by hydrogen bonds [N(4)–H(4A)···O(2), N(4)–H(4B)···O(1)#2, O(2)–H(2A)···O(1)#1] and formed a three-dimensional networks supramolecular structure (Figure S2, Table S2, Supplemental materials).

Luminescent Property

The fluorescence of 1, 2, 3, and L from solutions dissolved in methanol is shown in Figure 3; the fluorescence of 1, 2, 3, and L from solutions dissolved in DMF is shown in Figure 4.

Luminescent properties of compounds 1, 2, 3, and L were measured in methanol $(1.0 \times 10^{-5} \text{ mol} \cdot \text{L}^{-1})$ at room temperature (Figure 3). Upon the excitation of ultraviolet light, the emission peaks of 1 at 580 nm, 2 at 579 nm, and 3 at 581 nm, give yellow fluorescence,¹⁴ the emission peaks of L at 488 nm, which gives an intense blue luminescence in methanol.¹⁴ The result shows that the luminescence wavelength of L shifts to blue when compared with 1, 2, and 3 (π - π interaction and hydrogen bonds interaction in L).

1, 2, 3, and L have been dissolved in DMF $(1.0 \times 10^{-5} \text{ mol} \cdot \text{L}^{-1})$ at room temperature (Figure 4). Upon the excitation of ultraviolet light, the emission peaks of 1 at 569 nm, 2 at

	L	$[Cu(L_1)]$	
Empirical formula	$C_{13}H_{12}N_4O_2S$	$Cu(C_{12}H_{12}N_4O_2S)$	
Formula weight	288.34	339.86	
Temperature/K	123.3(2) K	113(2)	
Wavelength/Å	0.71073	0.71073	
Crystal system, space group	monoclinic, $P2_1/c$	monoclinic, $P2_1/c$	
Unit cell dimensions	$a = 7.9910(2) \text{ Å}; \alpha = 90^{\circ}$	$a = 9.600(13) \text{ Å}; \alpha = 90^{\circ}$	
	$b = 9.3310(3)$ Å; $\beta = 112.516(2)^{\circ}$	$b = 7.628(10) \text{ Å}; \beta =$ 97.90(3)°	
	$c = 19.5537(5)$ Å; $\gamma = 90^{\circ}$	$c = 18.26(2) \text{ Å}; \gamma = 90^{\circ}$	
Volume/Å ³	V = 1346.86(6)	V = 1324(3)	
Z, Calculated density/(Mg/m ³)	4, 1.422	4, 1.704	
Absorption coefficient/mm ⁻¹	0.247	1.812	
F(000)	600	692	
θ range for data collection	2.46°-25.01°	2.46°-25.01°	
Limiting indices	$-9 \leq h \leq 8, -10 \leq k \leq 11,$	$-11 \le h \le 11, -8 \le k \le 9,$	
	$-14 \leq 1 \leq 23$	$-20 \le l \le 21$	
Reflections collected/unique	4900/2375 [R(int) = 0.0186]	10255/2336 [R(int) = 0.1154]	
Completeness to θ	100.0%	100.00%	
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F ²	
Data/restraints/parameters	2375/0/193	2336/3/191	
Goodness-of-fit on F2	0.806	1.054	
Final <i>R</i> indices $[I > 2\sigma(I)]$	$R_1 = 0.0323, wR_2 = 0.0875$	R1 = 0.0670, wR2 = 0.1221	
<i>R</i> indices (all data)	$R_1 = 0.0376, wR_2 = 0.0941$	R1 = 0.0853, wR2 = 0.1332	
Largest diff. peak and hole/(e/nm ³)	219 and -226	552 and -693	

Table 1 Crystal data and structure refinement pa	arameters for L and $[Cu(L_1)]$
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568 nm, and **3** at 570 nm, give yellow fluorescence.¹⁴ The emission peaks of **L** at 475 nm, gives an intense blue luminescence in DMF,¹⁴ a little shifted toward blue compared to **1**, **2**, **3**, and **L** dissolved in methanol, which indicated that solvent slightly affects the emission peak wavelength.

L						
S(1)-C(13)	1.6822(17)	O(1)–C(2)	1.204(2)	N(3)-C(13)	1.360(2)	
O(2)–C(2)	1.368(2)	N(4)-C(13)	1.324(2)	N(3)–N(2)	1.368(2)	
O(2)–C(3)	1.404(2)	N(2)-C(12)	1.294(2)	C(1)–C(2)	1.494(3)	
N(1)-C(11)	1.333(2)	C(7)–C(9)	1.411(2)	C(7)–C(8)	1.429(2)	
N(1)-C(8)	1.366(2)	C(7)–C(6)	1.420(2)	C(12)–C(11)	1.470(2)	
C(6)–C(5)	1.362(3)	C(9)–C(10)	1.360(2)	C(4)–C(3)	1.361(2)	
C(4)–C(5)	1.414(3)	C(3)–C(8)	1.420(2)	C(11)-C(10)	1.417(2)	
C(2)–O(2)–C(3)	116.84(13)	N(2)-C(12)-C(11)	130.05(16)	O(1)-C(2)-C(1)	126.21(18)	
C(11)–N(11)–C(8)	117.50(13)	N(4)-C(13)-N(3)	116.48(15)	O(2)-C(2)-C(1)	111.24(16)	
C(13)–N(3)–N(2)	118.45(14)	C(4)-C(3)-O(2)	118.16(16)	N(3)-C(13)-S(1)	119.15(12)	
		$[Cu(L_1)]$				
Cu(1)–N(3)	1.889(5)	N(3)-Cu(1)-N(1)	89.19(18)	N(1)-Cu(1)-S(1)	156.63(14)	
Cu(1)–N(1)	1.943(5)	N(3)-Cu(1)-O(1)	172.64(15)	O(1)-Cu(1)-S(1)	114.85(11)	
Cu(1)–O(1)	1.956(4)	N(1)-Cu(1)-O(1)	85.15(17)			
Cu(1)-S(1)	2.439(3)	N(3)-Cu(1)-S(1)	69.77(13)			

Table 2 Selected bond lengths (Å) and angles (°) for L and $[Cu(L_1)]$



Figure 3 The fluorescence of the four compounds (1, 2, 3, and L) in methanol.

EXPERIMENTAL

Material and Methods

All the chemicals were obtained from commercial sources or prepared according to standard methods. Melting point was measured by an X4 Melting-Point Apparatus with a microscope and uncorrected. Elemental analysis was performed by a Perkin–Elmer 240



Figure 4 The fluorescence of the four compounds (1, 2, 3, and L) in DMF.

elementary analysis instrument. The ¹H NMR (500 and 400 MHz) was recorded on the Bruker AM-500 and Bruker AM-400 spectrometer. Chemical shifts (δ) are reported relative to TMS (¹H). IR spectra were recorded on a BIO-RAD FTS 3000. Mass spectra (ESI) were obtained on a Finnegan LCQ Advantage MAX spectrometer. Photoluminescence spectra were measured by a Cary Eclipse fluorescence spectrophotometer with a xenon arc lamp as the light source. The Supplemental materials contains additional synthetic procedures and ¹H NMR spectra for 2, 3, and L (Figures S3–S6). Compounds 2 and 3 were prepared according to reported procedures.^{11,12}

Synthesis of the 2-((*Z*)-Thiosemicarbazidomethyl)-Quinolin-8-yl Acetate (L)

An ethanolic solution (40 mL) of 2-formylquinolin-8-yl acetate (6 mmol, 1.29 g) was added drop by drop to the solution of thiosemicarbazide (6 mmol, 0.546 g) in ethanol (60 mL). The resulting mixture was refluxed for 4 h. The volume of solvent was reduced to 10 mL and the yellow precipitate was collected with filtration, washed with hot ethanol, then diethylether, the residue was recrystallized from the mixture of ethanol and acetone (2:1 v:v), and gave 1.2 g of yellow crystals L. Yield, 69%; M.p: 203.6–204.8 °C; IR (KBr): 3406 (N–H), 3039, 1761 (C=O), 1591 (C=N), 1532 (C–S–C), 1190, 1163 (C–O–C), 846(C=S) cm⁻¹; ¹H NMR (DMSO- d_6 , 500 MHz): $\delta = 13.95$ (s, 1H, NH), 8.76 (s, 1H, NH), 8.66 (dd, $J_1 = 8.3$, $J_2 = 3.8$ Hz, 1H), 8.42 (s, 1H), 7.96 (d, J = 8.1 Hz, 1H), 7.90 (dd, $J_1 = 8.6$, $J_2 = 2.5$ Hz, 1H), 7.73 (t, J = 7.8 Hz, 1H), 7.67 (d, J = 7.5 Hz, 1H), 7.60(s, 1H, CH), 2.67 (s, 3H, CH₃); ¹H NMR (DMSO- d_6 , D₂O, 500 MHz): $\delta = 8.61$ (d, $J_1 = 8.6$ Hz, 1H), 8.42 (s, 1H), 7.94 (dd, $J_1 = 8.2$, $J_2 = 1.1$ Hz, 1H), 7.84 (dd, J = 8.6 Hz, 1H), 7.71 (t, J = 7.9 Hz, 1H), 7.64 (dd, $J_1 = 7.6$, $J_2 = 1.2$ Hz, 1H), 7.57 (s, 1H, CH), 2.63 (s, 3H, CH₃); ESI-MS m/z(%): 311.1 [M + Na]⁺ (100%); Anal. Calcd (%) for C₁₃H₁₂N₄O₂S: C, 54.15; H, 4.20; N, 19.43; S, 11.12. found (%): C, 54.19; H, 4.23; N, 19.51; S, 11.16.

Preparation of the Complex [Cu(L₁)]

Dichloromethane (3 mL) was added drop by drop to a solution of $Cu(NO_3)_2 \cdot 3H_2O$ (3.65 mg, 0.0151 mmol) in H₂O (2 mL), and then, a solution of L (4.34 mg, 0.0151 mmol) in CH₃OH (4 mL) was added. After keeping the solution at room temperature for one week, black crystals were obtained by slow evaporation. IR (KBr): 3422(NH), 1633(C=N), 1488, 1445, 1387, 1336, 1075(C-O), 531(Cu-O). Anal. Calcd (%) for [Cu(L₁)]: C, 42.41; H, 3.56; N, 16.49; S, 9.43. found (%): C, 42.79; H, 3.32; N, 16.40; S, 9.52.

X-Ray Diffraction

A yellow crystal of **L** with dimensions of 0.20 mm \times 0.20 mm \times 0.20 mm was selected for single-crystal X-ray diffraction measurements preformed on a Super Nova detector. A black crystal of [Cu(L₁)] with dimensions of 0.20 mm \times 0.18 mm \times 0.12 mm was selected for single-crystal X-ray diffraction measurements preformed on Rigaku Saturn724 CCD detector. The data of **L** and [Cu(L₁)] were collected with graphite-monochromatic Mo *K*a radiation wavelength of 0.71073 Å using the $\omega/2\theta$ scan technique. Lorentz polarization and absorption corrections were applied. The structure was solved by direct methods and refined with a full-matrix least-square technique based on F^2 using the SHELXS-97¹⁵ and SHELXL-97¹⁶ programs. Anisotropic thermal parameters were assigned to all nonhydrogen atoms. All hydrogen atoms were generated geometrically. The crystal data and structure refinement details for $C_{13}H_{12}N_4O_2S$ (L) and [Cu(L₁)] are summarized in Table 1, and selected bond distances and angles are listed in Table 2. Hydrogen bond lengths and angles are located in Tables S1 and S2 (Supplemental materials).

Crystallographic data (**CIF**) for the structure reported in this article has been deposited in the Cambridge Crystallographic Data Centre as supplementary materials, CCDC No: 844459 for L; 860610 for [Cu(L₁)]. Copies of the data can be obtained, free of charge, on application to the CCDC, 12 Union Road, Cambridge CB12EZ, UK (Fax: ++44(1223) 336-033; email:deposit@ccdc.cam.ac.uk).

CONCLUSION

A new Schiff base 2-(thiosemicarbazide methyl) quinolin-8-yl acetate (**L**) and its Cu(II) complex [Cu(L₁)], have been synthesized and characterized. The stable threedimensional supramolecular structures of them were formed through intermolecular hydrogen bonds and/or π - π interactions between the molecules. From the data, figures and reference, it is said that **L** has a blue fluorescent emission at 488 in methanol and 475 nm in DMF solution at room temperature. If it coordinates with metals as the ligand, more light-emitting materials can be developed.¹⁷

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