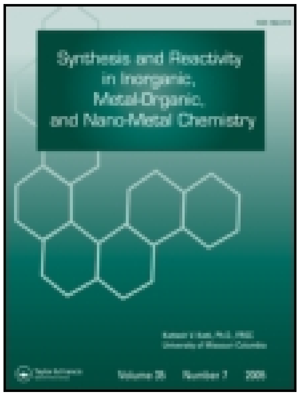

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One-Dimensional Chain Copper(II) and Nickel(II) Coordination Polymers With N-Salicylideneglycine Schiff Base Ligand

Shao-Ming Shi [a], Yun-Qiong Gu [a], Zhen-Feng Chen [a], Yan-Cheng Liu [a] & Hong Liang [a]
[a] Key Laboratory for the Chemistry and Molecular Engineering of Medicinal Resources (Ministry of Education of China), School of Chemistry and Chemical Engineering, Guangxi Normal University, Guilin, P. R. China

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Shao-Ming Shi, Yun-Qiong Gu, Zhen-Feng Chen, Yan-Cheng Liu, and Hong Liang
Key Laboratory for the Chemistry and Molecular Engineering of Medicinal Resources (Ministry of Education of China), School of Chemistry and Chemical Engineering, Guangxi Normal University, Guilin, P. R. China

[Cu(Sal-Gly)(Im)(H_2O)] (1) and [Ni(Sal-Gly)(H_2O)$_2$] (2) (Sal-Gly = N-salicylideneglycinate) was synthesized and structurally characterized. 1 and 2 are one-dimensional chain coordination polymers. In 1, Cu(II) ion has a five-coordinated square-pyramidal geometry and is surrounded by two O and one N atoms from a tetradentate Sal-Gly dianion and one N atom from imidazole ligand and one O from adjacent Sal-Gly ligand. In 2, Ni(II) adopts six-coordinated distorted octahedral geometry and is surrounded by two O and one N atoms from a tetradentate Sal-Gly dianion, two O from two aqua ligands, and one O from adjacent Sal-Gly ligand.

Keywords amino acid Schiff base, copper(II), crystal structure, nickel(II)

INTRODUCTION

The Schiff base transition metal complexes of amino acid have been the subject of considerable research because these complexes exhibit potential anticarcinogenic, antivirus, antibacterial, and antipyretic activities.[1–3] Recently, several reviews have focused on the supramolecular chemistry of Schiff base ligands.[4–9] Taking advantage of Schiff base to construct supramolecular structures have been attracted many chemists' interest. N-salicylideneglycinato-metal complexes are considered as the valuable nonenzymatic models of the action of Vitamin B6 enzyme.[10,11] In addition, a large number of amino acid Schiff base complexes have been investigated for their important biological properties including antibacterial activity,[12–13] scavenging effect on O_2,[14–16] and anticancer activity.[12] These results stimulated our research interest on the N-salicylideneglycinato-metal complexes. Although a series of N-salicylideneglycinato-metal complexes have been synthesized and characterized, such as nickel(II), copper(II), and zinc(II),[17–24] as well as oxovanadium(V) complexes,[25,26] the polymeric structures assembled by N-salicylideneglycine Schiff base and metal salts are rarely reported. Herein, we report the synthesis, crystal structure one-dimensional zigzag chain coordination polymers: [Cu(Sal-Gly)(Im)(H_2O)] (**1**) and [Ni(Sal-Gly)(H_2O)$_2$] (**2**) (Sal-Gly = N-salicylideneglycinate), which may enrich the supramolecular chemistry of Schiff base.

EXPERIMENTAL

Materials

All chemicals were purchased from commercial sources and used without further purification. N-salicylideneglycine Schiff base were prepared by literature method.[27]

Physical Measurements

C, H, and N data were obtained using Perkin-Elmer 2400II CHNS/O elemental analyzer (Waltham, MA, USA). Infrared spectra were measured on a Nicolet FT-IR spectrophotometer (Madison, WI, USA) with KBr pellets in the range 4000–400 cm^{-1}. X-ray single crystal structural analyses were performed on a Bruker CCD area detector diffractometer (Germany) with graphite-monochromatized Mokα radiation.

Synthesis of [Cu(Sal-Gly)(Im)] (1)

To a solution of $Cu(CH_3COO)_2{\cdot}H_2O$ (0.200 g, 1 mmol) in 95% ethanol (20 mL) was added to N-salicylideneglycine Schiff base(Sal-Gly) (0.179 g, 1 mmol) and imidazole (Im) (0.068 g, 1 mmol) in 95% ethanol (20 mL). The mixture was stirred at 60–70°C for 5 h. The precipitates was collected by filtration, washed successively with water and ethanol, and dried in air.

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Address correspondence to Zhen-Feng Chen, Key Laboratory for the Chemistry and Molecular Engineering of Medicinal Resources (Ministry of Education of China), School of Chemistry and Chemical Engineering, Guangxi Normal University, Guilin 541004, P. R. China. E-mail: chenzfgxnu@yahoo.com

TABLE 1
Crystallographic data and refinement details for **1** and **2**

	1	**2**
Formula	$C_{12}H_{11}CuN_3O_3$	$C_{18}H_{22}N_2Ni_2O_{10}$
Formula weight	308.78	543.80
Crystal system	monoclinic	monoclinic
Space group	*P*2(1)/c	*P*2(1)/c
a (Å)	11.057(4)	15.369(2)
b (Å)	11.509(5)	7.4345(11)
c (Å)	9.676(4)	9.1435(13)
β (°)	103.141(4)	106.532(3)
V (Å^3)	1199.1(8)	1001.6(3)
Z	4	2
D_c (g/cm$^{3)}$)	1.710	1.803
μ (mm^{-1})	1.828	1.942
Crystal size (mm^3)	0.31 × 0.27 × 0.15	0.28 × 0.24 × 0.22
F(000)	628	560
R_1 [I > 2σ(I)	0.0334	0.0291
wR_2 (all data)	0.0908	0.0836

Then, a portion of precipitate was dissolved in aqueous ethanol (H_2O:CH_3CH_2OH = 1:2, 30 mL), and it was maintained at ambient temperature for slow evaporation. After seven days, a dark-green block crystals of **1** suitable for X-ray diffraction analysis were isolated in 30% yield. Anal. Calcd. for $C_{12}H_{11}CuN_3O_3$ (%): C, 46.68; H, 3.59; N, 13.61. Found (%): C, 46.52; H, 3.60; N, 13.70. Main IR (KBr, cm^{-1}): 3025(w), 1642(s), 1576(s), 1453(ms), 1383(ms), 760(w), 657(w), 533(w).

Synthesis of [Ni(Sal-Gly)(H_2O)$_2$] (2)

This complex was prepared in a similar process as for **1**. $Ni(CH_3COO)_2 \cdot 4H_2O$ was used to replace $Cu(CH_3COO)_2 \cdot H_2O$, but without imidazole. A bright green block crystals of **2** suitable for X-ray diffraction analysis was obtained in 40% yield. Anal. Calcd. for $C_{18}H_{22}N_2Ni_2O_{10}$ (%): C, 39.76; H, 4.08; N, 5.15. Found (%): C, 39.61; H, 4.12; N, 5.06. Main IR (KBr, cm^{-1}): 3369(m), 1657(s), 1599(s), 1476(ms), 1297(ms), 1195.1(m), 748(m), 523(w).

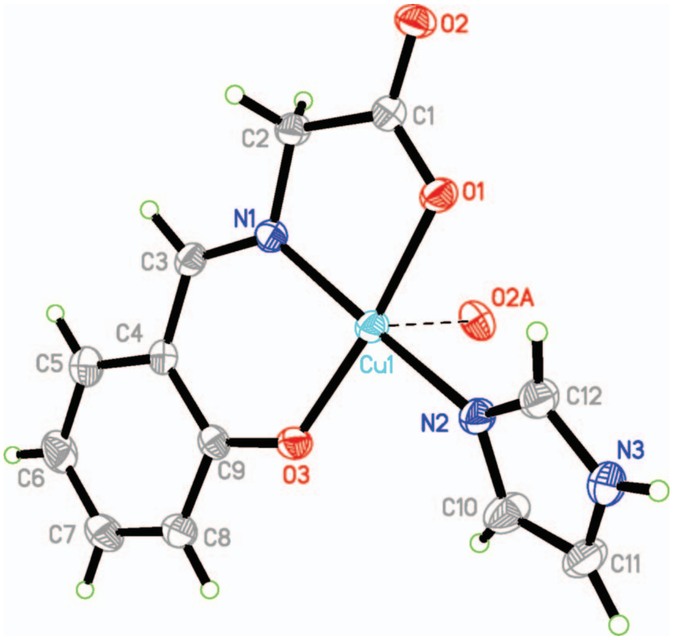

FIG. 1. ORTEP view of **1** with 30% thermal ellipsoids showing the atom-labeling around copper(II). Symmetry transformations used to generate equivalent atoms, A: x, 1.5 − y, 0.5 + z (color figure available online).

X-Ray Studies

The crystals of **1** and **2** were selected and mounted on a glass fiber in air. The diffraction data were collected on a Bruker CCD diffractometer (Germany) equipped with graphite monochromated Mo-Kα radiation (λ = 0.71073Å, 50 kV, 150 mA) using the Φ-ω scan mode at room temperature. Raw frame data were integrated with the SAINT program (University of Göttingen, Göttingen, Germany).[28] The structures were solved by direct methods using SHELXS-97 and refined by full-matrix least squares using SHELXL-97 (Bruker AXS Inc., Madison, WI, USA).[29] An empirical absorption correction was applied with SADABS (University of Göttingen, Göttingen, Germany).[30] All non-hydrogen atoms were refined anisotropically and hydrogen atoms were located in the calculated positions and refined by a riding mode, with a common thermal parameter. The drawings were made with XP and Diamond, and all calculations were performed on a PC computer with SHELXTL (Bruker AXS Inc.,

TABLE 2
Selected bond length (Å) and angles (°) for **1**

Cu(1)−O(1)	1.989 (2)	Cu(1)−N (1)	1.927 (2)
Cu(1)−O(2)	1.918 (2)	Cu(1)−N(2)	1.958 (2)
Cu(1)−O(2A)	2.5260 (7)		
O(1)−Cu(1)−N(1)	83.39 (9)	O(1)−Cu(1)−N(2)	92.47 (9)
O(1)−Cu(1)−O(3)	171.50 (9)	N(1)−Cu(1)−N(2)	172.69 (10)
O(3)−Cu(1)−N(1)	97.75 (9)	O(3)−Cu(1)−N(2)	90.53 (9)

Symmetry transformations used to generate equivalent atoms, A: x, 1.5 − y, 0.5 + z.

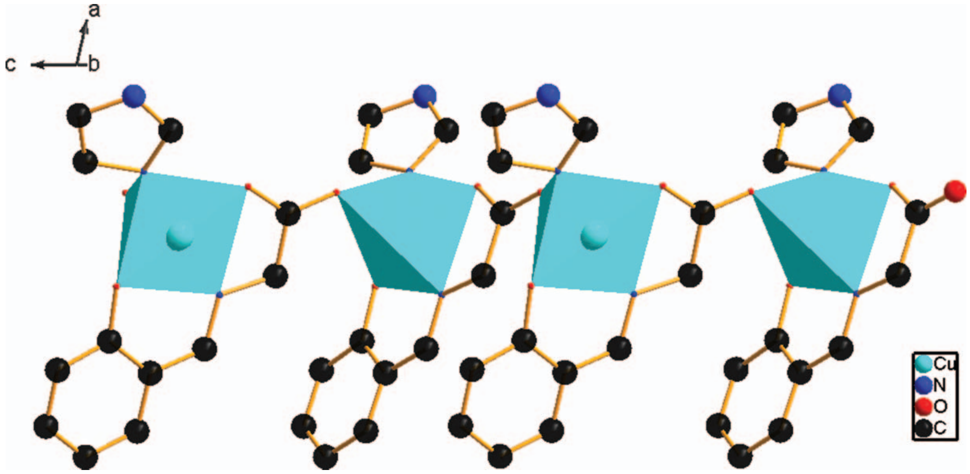

FIG. 2. One-dimensional zigzag chain with polyhedron geometry around copper(II) centers. For clarity, H atoms were omitted (color figure available online).

Madison, WI, USA).[29] The crystal data, data collection and refinement details are summarized in Table 1.

RESULTS AND DISCUSSION

Synthesis and Characterization of the Complexes

Reactions of Sal-Gly with Cu(II) (at presence of imidazole) and Ni(II) salts gave complexes **1** and **2**. The IR spectra provided valuable information regarding the nature of functional groups present in the ligands and their metal complexes. The strong peaks at 1612 cm^{-1} attributed to C=N stretching vibration band of Sal-Gly ligand are shifted to low frequency in complexes **1** and **2** (1576 and 1599 cm^{-1}), which indicate that the nitrogen of the imine group is coordinated to the metal ions.[31–33] The broad bands in the range 3050–3075 cm^{-1} ascribed to intramolecular H-bonded –OH groups of Sal-Gly ligand,[34] which are not present in complexes **1** and **2**, indicating that the oxygen of the –OH groups are coordinated to the metal ions.[31] The characteristic stretching vibrations of free carboxyl group (Sal-Gly) at 1730 cm^{-1} are split into two peaks: 1647 [$\upsilon_{as}(COO^-)$] and 1453 cm^{-1} [$\upsilon_s(COO^-)$] for **1** with $\Delta\upsilon = 194$ cm^{-1}; 1657 and 1476 cm^{-1} for **2** with $\Delta\upsilon = 181$ cm^{-1}. The splitting of $\upsilon_{as}(COO^-)$ and $\upsilon_s(COO^-)$ indicates that the oxygen of carboxylate of Sal-Gly is coordinated to the metal ion.

Structural of [Cu(Sal-Gly)(Im)] (1)

The molecular structure of [Cu(Sal-Gly)(Im)] (**1**) with the atom-numbering scheme is illustrated in Figure 1 and selected bond distances and angles are listed in Table 2. As shown in Figure 1, the central metal atom Cu1 coordinates by three oxygens and one N from two tetradentate Schiff base (Sal-Gly) and one nitrogen from imidazole, thus forms a square-pyramidal geometry, which is similar to (imidazole)(N-salicylidene-beta-alaninato)copper(II),[35] [Cu(Sal-Ser)(2-amino pyridine)] (Sal-Ser = N-salicylidene-L-serinate).[36] The N(1), O(1), O(3), N(2) composes of the bottom plane of the square-pyramid with common bond distances (Cu(1)–O(1) 1.989(2), Cu(1)–O(2) 1.918(2), Cu(1)–N(1) 1.9313(18), and Cu(1)–N(2) 1.9619(18) Å), which are well comparable with those found for the Cu(II) complexes derived from salicyladehyde and amino acid—(imidazole) (N-salicylidene-beta-alaninato)copper(II),[35] [Cu(Sal-Ser)(2-amino pyridine)][36]—while a weakly coordinated O(2A) from an adjacent complex unit is located on the apical site with Cu–O(2A) distance of 2.5260(7) Å. In addition, The Cu(1)–O bond length to phenolic oxygen O(1) of the tridentate Schiff base is significantly shorter than the bonding to carboxylic oxygen O(2), which indicates different bond strengths.[35] By means of the bidentate bridge COO$^-$ of Sal-Gly, the adjacent copper(II) atoms are linked, resulting in a one-dimensional zigzag chain (Figure 2). It should be pointed out that the hydrogen bond between the imidazole and O(2) also attributable to the zigzag conformation.

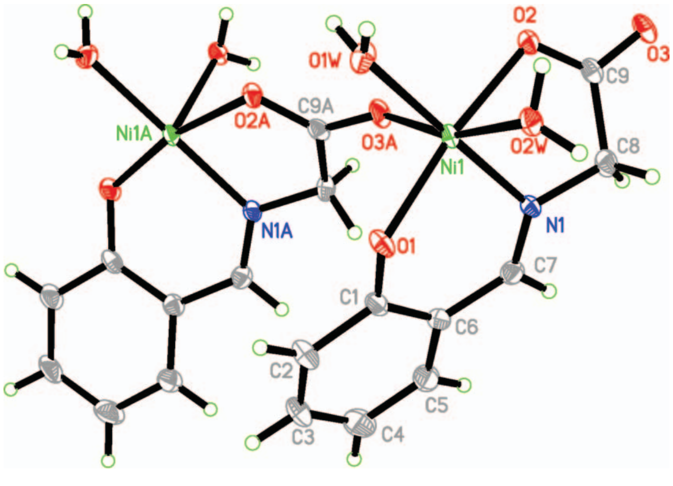

FIG. 3. ORTEP view of **2** with 30% thermal ellipsoids showing the atom-labeling around nickel(II). Symmetry transformations used to generate equivalent atoms, A: x, 0.5 − y, −0.5 + z (color figure available online).

TABLE 3
Selected bond length (Å) and angles (°) for **2**

Ni(1)–N(1)	1.994 (2)	Ni(1)–O(1W)	2.027 (2)
Ni(1)–O(3A)	2.1294 (19)	Ni(1)–O(1)	2.0209 (19)
Ni(1)–O(2)	2.1030 (19)	Ni(1)–O(W)	2.1384 (19)
N(1)–Ni(1)–O(1)	89.13 (9)	O(1)–Ni(1)–O(2)	168.57 (7)
N(1)–Ni(1)–O(2)	80.22 (9)	N(1)–Ni(1)–O(3)	92.81 (9)
N(1)–Ni(1)–O(1W)	91.92 (9)	O(1W)–Ni(1)–O(3)	85.81 (8)
N(1)–Ni(1)–O(2W)	96.26 (8)	O(1W)–Ni(1)–O(2W)	84.87 (8)
O(1W)–Ni(1)–O(2)	98.63 (8)	O(1)–Ni(1)–O(3)	92.76 (8)
O(2)–Ni(1)–O(3)	83.70 (7)	O(1)–Ni(1)–O(2W)	100.18 (8)
O(2)–Ni(1)–O(2W)	85.21 (7)	O(3)–Ni(1)–O(2W)	164.27 (8)

Symmetry transformations used to generate equivalent atoms, A: x, 0.5 – y, –0.5 + z.

TABLE 4
Hydrogen bonds for **2**

	D–H	H···A	D···A	∠(DHA)(°)
O(1W)–H(1WA)···O(3)#3	0.84	1.96	2.773 (3)	163.7
O(1W)–H(1WB)···O(2W)#4	0.84	1.98	2.815 (3)	176.7
O(2W)–H(2WB)···O(2)#3	0.83	1.96	1.783 (3)	170.0
O(2W)–H(2WA)···O(1)#5	0.84	1.85	2.679 (3)	169.5

Symmetry transformations used to generate equivalent atoms, #3: −x + 1, −y, −z + 1; #4: −x + 1, y + 1/2, −z + 1/2; #5: x, −y − 1/2, z + 1/2.

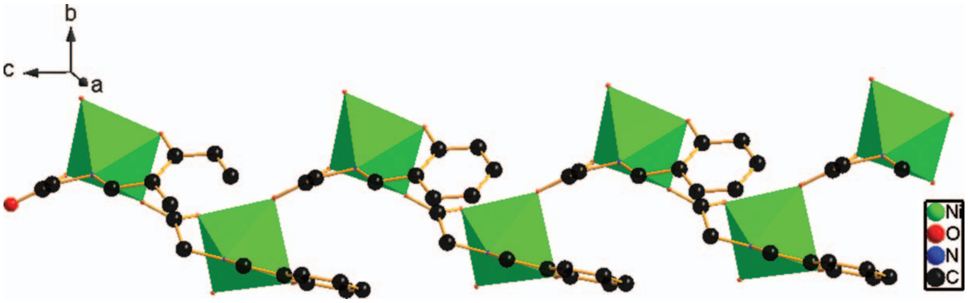

FIG. 4. One-dimensional zigzag chain with polyhedron geometry around nickel(II) centers. For clarity, H atoms were omitted (color figure available online).

Structural of [Ni(Sal-Gly)(H_2O)$_2$] (2)

The molecular strcutrue of [Ni(Sal-Gly)(H_2O)] (**2**) with the atom-numbering scheme is shown in Figure 3 and selected bond distances and angles are listed in Table 3. The metal center Ni(II) adopts six-coordinated distorted octahedral geometry and is surrounded by one N and two O atoms from the same Sal-Gly ligand, two aqua ligands, and to one O from another Sal-Gly ligand. Similar to complex **1**, the Sal-Gly ligand is tetradentate. A one-dimensional zigzagic chain (Figure 4) is formed through the bidentate bridge COO^- of Sal-Gly to link the adjacent copper(II) atoms. The bond distances and angles are in normal range, which are compared to those Sal-Gly nickel(II) complex ($C_9H_7NO_3$)Ni($C_3H_4N_2$)$_3$·H_2O ($C_9H_7NO_3$ = Sal-Gly, $C_3H_4N_2$ = pyrazole).[16] In addition, three dimensional network is generated via hydrogen bonding involving aqua and carboxylic group (hydrogen bonds are listed in Table 4).

SUPPLEMENTARY MATERIALS

Crystallographic data for the crystal structures of **1** and **2** reported in this article have been deposited with the Cambridge Crystallographic Data Center (CCDC No. 835882, 835883), respectively. These materials can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: t44 1223 336033; Email: deposit@ccdc.cam.ac.uk).

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