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肟型 Schiff 碱 Cu(Ⅱ)和 Ni(Ⅱ)配合物的合成、晶体结构及光谱性质

郭建强 孙银霞* 俞 彬 李 璟 贾浩然
(兰州交通大学化学与生物工程学院,兰州 730070)

摘要：合成了 2 个 Schiff 碱 Cu(Ⅱ)/Ni(Ⅱ)配合物[Cu(L^1)$_2$] (**1**)和[Ni(L^2)$_2$] (**2**)(HL^1=1-(4-(((*E*)-3,5-dichloro-2-hydroxybenzylidene)amino)henyl)ethanoe *O*-benzyloxime,HL^2=1-(4-(((*E*)-4-methoxy-2-hydroxybenzylidene)amino)phenyl)ethanone *O*-benzyloxime),并通过元素分析、红外光谱、紫外光谱及 X 射线单晶衍射分析进行了表征和分析。X 射线结构表明:配合物 **1** 和 **2** 具有类似的结构,均由 1 个金属离子和 2 个配体单元组成。配合物 **1** 和 **2** 都是单斜晶系,但配合物 **1** 空间群为 *C*2/*c*,而配合物 **2** 为 $P2_1/c$。且中心金属 Cu(Ⅱ)和 Ni(Ⅱ)离子的空间构型均为四配位的扭曲的平面四边形结构。配合物 **1** 通过 $\pi\cdots\pi$ 和 C–H$\cdots\pi$ 作用形成 3D 超分子结构,而配合物 **2** 通过 C–H$\cdots\pi$ 作用形成 2D 超分子孔道结构。

关键词：铜(Ⅱ)配合物；镍(Ⅱ)配合物；晶体结构；Schiff 碱配体；光谱性质
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Syntheses, Crystal Structures and Spectroscopic Properties of Copper(Ⅱ) and Nickel(Ⅱ) Complexes with Oxime-Type Schiff Base Ligands

GUO Jian-Qiang　SUN Yin-Xia*　YU Bin LI Jing　JIA Hao-Ran
(*School of Chemical and Biological Engineering, Lanzhou Jiaotong University, Lanzhou* 730070, *China*)

Abstract: Two copper(Ⅱ) and nickel(Ⅱ) complexes, [Cu(L^1)$_2$] (**1**) (HL^1=1-(4-(((*E*)-3,5-dichloro-2-hydroxybenzylidene)amino)henyl)ethanoe *O*-benzyloxime) and [Ni (L^2)$_2$] (**2**) (HL^2 =1-(4-(((*E*)-4-methoxy-2-hydroxybenzylidene)amino)phenyl)ethanone *O*-benzyloxime), have been synthesized and characterized by elemental analyses, IR and UV-Vis spectra and X-ray single crystal diffraction method. X-ray structure showed that complexes **1** and **2** have the similar structure, consisting of one metal ion, two ligand units. But the complex **1** crystallize in the monoclinic space group *C*2/*c* with the unit cell parameters: *a*=2.718 77(13) nm, *b*=1.321 99(6) nm, *c*=2.225 56(12) nm, β=92.069 0 (10)°; whereas the complex **2** crystallize in the monoclinic space group $P2_1/c$, with the unit cell parameters: a=1.423 89(18) nm, *b*=0.601 22(8) nm, *c*=2.467 7(2) nm, β=113.353(2)°. The center Cu(Ⅱ) or Ni(Ⅱ) atoms are all four-coordinated in a *trans*-N_2O_2 slightly distorted square-planar geometry by two phenoxy O atoms and two imine N atoms from two symmetry-related N,O-bidentate Schiff base ligands. And the supramolecular structures of the two complexes **1** and **2** are widely different by obvious intermolecular interaction. The complex **1** links some other molecules into an infinite 3D layer supramolecular structure via intermolecular C–H$\cdots\pi$ and $\pi\cdots\pi$ stacking interactions between neighboring benzene rings, while complex **2** formed 2D supramolecular channel structure by intermolecular C–H$\cdots\pi$ hydrogen bond interactions. CCDC: 1560091, **1**; 1560090, **2**.

Keywords: Cu(Ⅱ) complex; Ni(Ⅱ) complex; crystal structure; Schiff-base ligand; spectroscopic property

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兰州交通大学研究生教改项目(No.160012)资助。
*通信联系人。E-mail:sun_yinxia@163.com

0 Introduction

The coordination chemistry of transition metal complexes with Schiff base ligands has achieved a considerable attention in the last decades[1-5], because of their redox-chemistry, unusual magnetic and structural properties, as well as their usage as models for metalloproteinase[6-10], and catalysts for oxidation and polymerization reactions[6,8,11]. It is important to introduce suitable functional groups into the organic moiety of the ligands in order to improve or tune the properties of these metal complexes[12-16]. Oxime-containing Schiff base ligands as a class of strong electron donors, and it is found that they can efficiently stabilize high oxidation states of metal ions, such as Cu(Ⅱ) and Ni(Ⅱ)[17-20]. Herein, in order to further study the supramolecular structures of the transition metal complexes with the oxime-type Schiff base ligands, two supramolecular Cu(Ⅱ) and Ni(Ⅱ)complexes with oxime-type Schiff base ligands, $[Cu(L^1)_2]$ (**1**) (HL^1=1-(4-(((*E*)-3, 5-dichloro-2-hydroxybenzylidene)amino)phenyl)ethanoe *O*-benzyloxime), and $[Ni(L^2)_2]$ (**2**) (HL^2=1-(4-(((*E*)-4-methoxy-2-hydroxybenzylidene)amino)phenyl)ethanone *O*-benzyloxime), have been synthesized and characterized by elemental analyses, IR spectra, UV-Vis spectra and X-ray crystallographic analysis.

1 Experimental

1.1 Materials

4-aminoacetophenone, *O*-benzylhydroxylamine, 4-methoxysalicylaldehyde, and 3,5-dichloro-2-hydroxybenzaldehyde were purchased from Aldrich and used without further purification. The other reagents and solvents were analytical grade reagents from Tianjin Chemical Reagent Factory.

1.2 Methods

C, H and N analyses were carried out with a GmbH VariuoEL V3.00 automatic elemental analyzer. FT-IR spectra were recorded on a VERTEX70 FT-IR spectrophotometer, with samples prepared as KBr (400~4 000 cm^{-1}). UV-Vis absorption spectra were recorded on a Shimadzu UV-2550 spectrometer. X-ray single crystal structure was determined on a Bruker Smart 1000 CCD area detector. Melting points were measured by a microscopic melting point apparatus made in Beijing Taike Instrument Limited Company and the thermometer was uncorrected.

1.3 Synthesis of HL^1 and HL^2

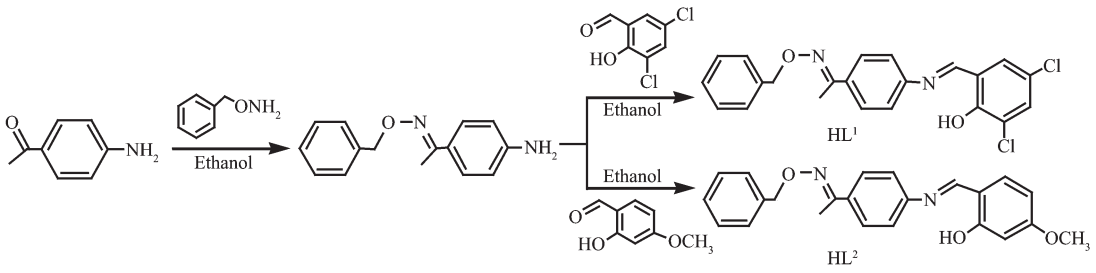
HL^1 and HL^2 were synthesized according to the reported method[21-22]. The synthetic route is given in Scheme 1. HL^1: Yield, 81.6%; m.p. 399~400 K. Anal. Calcd. for $C_{23}H_{21}Cl_2N_2O_2$ (%): C, 64.49; H, 4.94; N, 6.54; Found(%): C, 64.51; H, 4.92; N, 6.57. HL^2: Yield, 79.6%; m.p. 399~401 K. Anal. Calcd. for $C_{23}H_{22}N_2O_3$ (%): C, 73.78; H, 5.92; N, 7.48; Found(%): C,73.75; H, 5.90; N, 7.50.

1.4 Synthesis of $[Cu(L^1)_2]$ (1)

A methanol solution (5 mL) of Cu(Ⅱ) acetate monohydrate (1.90 mg, 0.01 mmol) was added dropwise to an acetone solution (3 mL) of HL^1 (8.26 mg, 0.01 mmol) at room temperature. The mixing solution turned to red-brown immediately, was filtered and the filtrate was allowed to stand at room temperature for about two weeks. Brown block-shaped single crystals suitable for X-ray structural determination were obtained. Anal. Calcd. for $C_{44}H_{34}Cl_4CuN_4O_4$(%): C, 59.51; H, 3.86; N, 6.31. Found(%): C, 60.02; H, 3.73; N, 6.55.

1.5 Synthesis of $[Ni(L^2)_2]$ (2)

A solution of Ni(Ⅱ) acetate tetrahydrate (1.89 mg,



Scheme 1 Synthetic routes of HL^1 and HL^2

0.01 mmol) in acetonitrile (4 mL) was added dropwise to a solution of HL^2 (7.49 mg, 0.01mmol) in *n*-propanol (3 mL) at room temperature. The color of the mixing solution turned green immediately. The mixture solution was filtered and the filtrate was allowed to stand at room temperature for about three days, the solvent was partially evaporated and obtained several green prismatic single crystals suitable for X-ray crystallographic analysis. Anal. Calcd. for $C_{46}H_{42}N_4NiO_6$ (%): C, 68.59; H, 5.26; N, 6.96. Found(%): C, 69.05; H, 5.06; N, 6.99.

1.6 Crystal structure determination

The single crystals of complexes **1** and **2** with approximate dimensions of 0.30 mm×0.27mm×0.15 mm and 0.21 mm×0.14 mm×0.08 mm were placed on a Bruker Smart 1000 CCD area detector. The diffraction data were collected using a graphite monochromated Mo $K\alpha$ radiation (λ=0.071 073 nm) at 293(2) K for complex **1** and a graphite monochromated Cu $K\alpha$ radiation (λ=0.154 178 nm) at 293(2) K for complex **2**. Empirical absorption correction was applied to the data using SADABS program[23]. The structures were solved by direct methods and refined by full-matrix leastsquares method on F^2 using the SHELXL-97 program[24]. All nonhydrogen atoms were refined anisotropically. All the hydrogen atoms were generated geometrically and refined isotropically using the riding model. Details of the crystal parameters, data collection and refinements for complexes **1** and **2** are summarized in Table 1.

CCDC: 1560091, **1**; 1560090, **2**.

2 Results and discussion

2.1 IR spectra analyses

The FT-IR spectra of HL^1, HL^2 and its corresponding complexes **1** and **2** exhibit various bands in the 400~4 000 cm^{-1} region in Table 2. The free ligand HL^1 and HL^2 exhibit characteristic C=N stretching bands at 1 599 and 1 626 cm^{-1}, while the C=N of complex **1** and complex **2** are observed in the 1 595

Table 1　Crystal data and structure refinement for complexes 1 and 2

Complex	1	2
Empirical formula	$C_{44}H_{34}Cl_4CuN_4O_4$	$C_{46}H_{42}NiN_4O_6$
Formula weight	888.10	805.53
Crystal system	Monoclinic	Monoclinic
Space group	*C*2/*c*	$P2_1/c$
a / nm	2.718 77(13)	1.423 89(18)
b / nm	1.321 99(6)	0.601 22(8)
c / nm	2.225 56(12)	2.467 7(2)
β / (°)	92.069(1)	113.353(2)
V / nm^3	7.993 9(7)	1.939 5(4)
Z	8	2
D_c / ($g\cdot cm^{-3}$)	1.476	1.379
μ / mm^{-1}	0.863	1.180
F(000)	3 640	844
θ range for data collection / (°)	2.53~25.02	3.38~66.02
Limiting indices	$-32\leqslant h\leqslant 22$; $-15\leqslant k\leqslant 15$; $-26\leqslant l\leqslant 24$	$-16\leqslant h\leqslant 16$; $-7\leqslant k\leqslant 6$; $-21\leqslant l\leqslant 29$
Reflection collected, unique	26 167, 7 052 (R_{int}=0.077 0)	5 545, 3 376 (R_{int}=0.051 9)
Completeness to θ / %	99.9%(θ=25.02°)	99.9%(θ=66.02°)
Data, restraint, parameter	7 052, 0, 516	3 376, 0, 21
GOF on F^2	1.014	1.010
Final *R* indices [$I>2\sigma(I)$]	R_1=0.054 0, wR_2=0.100 9	R_1=0.072 5, wR_2=0.154 0
R indices (all data)	R_1=0.104 2, wR_2=0.124 3	R_1=0.135 0, wR_2=0.214 1
$(\Delta\rho)_{max}$, $(\Delta\rho)_{min}$ / ($e\cdot nm^{-3}$)	628, −326	302, −616

Table 2 IR spectrum of HL^1, HL^2 and theirs corresponding Cu(Ⅱ) and Ni(Ⅱ) complexes

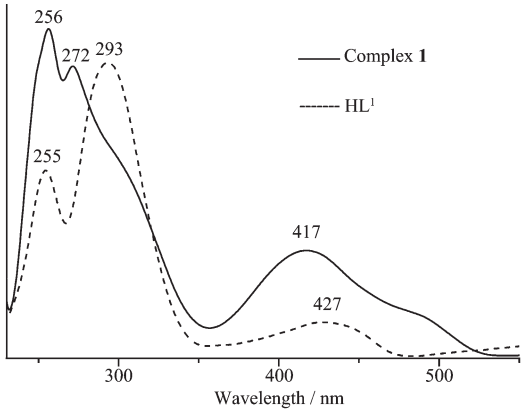
cm^{-1}

	ν(C=N)	ν(Ar-O)	ν(Cu-N)	ν(Cu-O)	ν(C=C)	Benzene ring	Skeleton
HL^1	1 599	1 171	—	—	1 564	1 490	1 452
Complex **1**	1 595	1 169	540	503	1 531	1 500	1 445
HL^2	1 626	1 206	—	—	1 589	1 543	1 483
Complex **2**	1 649	1 213	519	463	1 548	1 526	1 456

and 1 649 cm^{-1}, respectively. The C=N stretching frequencies shift to lower frequencies by *ca.* 17 and 18 cm^{-1} upon complexation respectively, indicating a decrease in the C=N stretching bond order due to the coordinated bond of the metal atom with the imino nitrogen lone pair[25]. In the 1 445~1 564 cm^{-1} region, the observed bands are attributed to aromatic C=C vibrations. Upon coordination these bands shift to lower frequencies for the metal complexes[26]. The Ar-O stretching frequency appears as a strong band at 1 171, 1 206 cm^{-1} for HL^1 and HL^2, and at 1 169, 1 213 cm^{-1} for the complexes, respectively[27]. The Ar-O stretching frequency shifts to lower frequency, indicating that the M-O bond forms between the metal atoms and oxygen atoms of the phenolic groups[5]. The far-infrared spectra of complexes **1** and **2** was also obtained in the region 500~100 cm^{-1} in order to identify frequencies due to the M-O and M-N bonds. The FT-IR spectrum of complex **1** shows (M-N) and (M-O) vibration absorption frequencies at 540 and 503 cm^{-1} (or 519 and 463 cm^{-1} for complex **2**), respectively. These assignments are consistent with the literature frequency values[28].

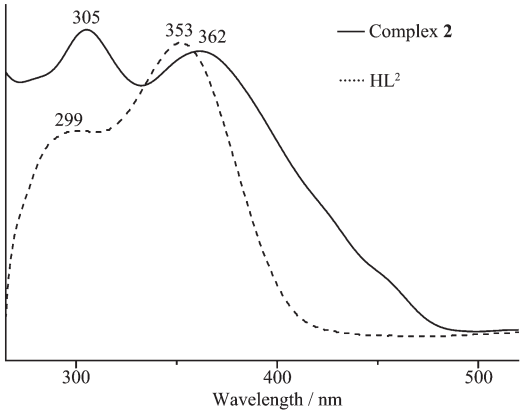
2.2 UV-Vis absorption spectra analyses

The absorption spectra of ligand HL^1, HL^2 and theirs corresponding Cu(Ⅱ) and Ni(Ⅱ) complexes were determined in diluted DMF solution as shown in Fig.1 and 2. UV-Vis spectrum of the free ligand HL^1 exhibits three absorption peaks at *ca.* 255, 293 and 427 nm (Fig.1). The absorption peak of HL^1 at 255 nm assigned to the π-π^* transition of the benzene rings shifts to low energy region by *ca.* 1 nm in complex **1**, indicating Cu(Ⅱ) ion coordinated with the O and N atoms of ligand units. The absorption peak of HL^1 at 293 and 427 nm attributed to the intra-ligand π-π^* transition of the C=N bonds shift to 272 and 417 nm in complex **1** upon complexation, respectively. The UV-Vis spectrum of the free ligand HL^2 exhibits two absorption peaks at *ca.* 299 and 353 nm. The former absorption peak at 299 nm can be assigned to the π-π^* transition of the benzene rings and the latter one at 353 nm can be attributed to the intra-ligand π-π^* transition of the C=N bonds[29]. Compared with the



$c=5\times10^{-5}$ mol·L^{-1}

Fig.1 UV-Vis absorption spectra of HL^1 and Cu(Ⅱ) complex in dilute DMF solution at room temperature



$c=5\times10^{-5}$ mol·L^{-1}

Fig.2 UV-Vis absorption spectra of HL^2 and Ni(Ⅱ) complex in dilute DMF solution at room temperature

absorption peak of the free ligand HL^2, a corresponding absorption peak at 291 nm is observed in the Ni(Ⅱ) complex, which bathochromically shifts for *ca.* 6 nm, indicating the coordination of Ni(Ⅱ) ions with the free ligand. Meanwhile, the absorption band at about 353 nm disappears from the UV-Vis spectrum of the Ni(Ⅱ) complex, which indicates that the oxime nitrogen atom is involved in the coordination to the Cu(Ⅱ) atom[30-31]. In addition, the new bands observed at 362 nm for the Ni(Ⅱ) complex is assigned to the n-π^* charge transfer transition from the filled $p\pi$ orbital of the phenolic oxygen to the vacant-orbital of the Ni(Ⅱ) ions, which are characteristic of the transition metal complexes with N_2O_2 coordination spheres[32].

2.3 Crystal structures of the Cu(Ⅱ) and Ni(Ⅱ) complexes

The single crystal structures of complexes **1** and **2** were confirmed by X-ray crystallography (Fig.3 and 4). The selected bond lengths and angles of complexes **1** and **2** are listed in Table 3. X-ray crystallographic analysis reveals that both complexes **1** and **2** crystallize in the monoclinic system with space group *C*2/*c* for complex **1** and $P2_1/c$ for **2**. The complexes **1** and **2** can be described as mononuclear M(Ⅱ) complexes (M= Cu or Ni), consist of one M(Ⅱ) ion and two L^- units, in which the M(Ⅱ) ion is four-coordinated in a *trans*-N_2O_2 square-planar geometry, with two phenolic O and two imino N atoms from two N,O-bidentate oxime-type ligands (HL^1 and HL^2).

The crystal structure of the complex **1** is stabilized by four intramolecular non-classic hydrogen bonds (Fig.5, Table 4). Meanwhile, the Cu(Ⅱ) complex is linked by $\pi\cdots\pi$ stacking interactions (Fig.6, Table 5) between benzene rings or/and chelate ring of the adjacent Cu(Ⅱ) complex molecules into an infinite 3D

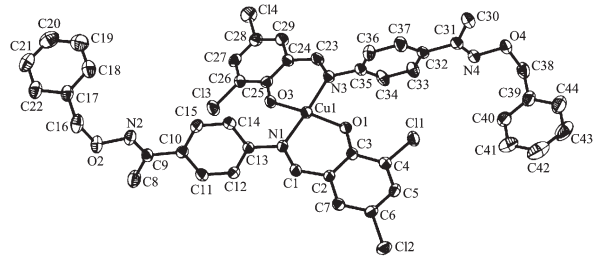
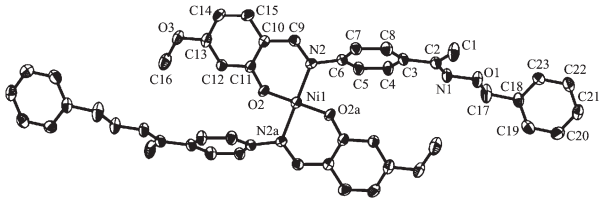


Fig.3　Molecular structure of complex **1** showing 30% probability displacement ellipsoids



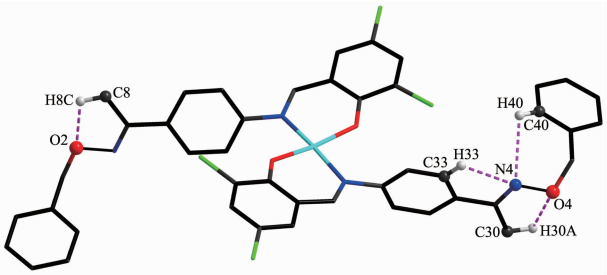
Symmetry codes: a: 1–*x*, 1–*y*, 1–*z*

Fig.4　Molecular structure of complex **2** showing 30% probability displacement ellipsoids

Table 3　Selected bond lengths (nm) and bond angles (°) for complexes 1 and 2

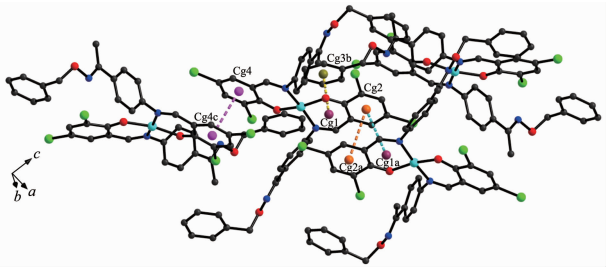
Complex **1**					
Cu1-O1	0.188 9(3)	Cu1-O3	0.190 1(3)	Cu1-N1	0.199 7(3)
Cu1-N3	0.199 7(3)				
O1-Cu1-O3	168.012)	O1-Cu1-N1	92.8(1)	O1-Cu1-N3	87.7(1)
O3-Cu1-N1	89.3(1)	O3-Cu1-N3	91.7(1)	N1-Cu1-N3	172.7(1)
Cu1-O1-C3	128.8(2)	Cu1-O3-C25	130.2(3)	Cu1-N3-C35	122.4(3)
Cu1-N1-C1	123.3(3)	Cu1-N1-C13	122.0(3)	Cu1-N3-C23	123.7(3)
Complex **2**					
Ni1-O2	0.182 5(5)	Ni1-N2	0.191 9(4)	Ni1-O2a	0.182 5(5)
Ni1-N2a	0.191 9(4)				
O2-Ni1-N2	91.54(18)	O2-Ni1-O2a	180.00	O2-Ni1-N2a	88.46(18)
N2-Ni1-O2a	88.46(18)	N2-Ni1-N2a	180.00	O2a-Ni1-O2a	91.54(18)
Ni1-O2-C11	125.7(4)	Ni1-N2-C6	117.7(3)	Ni1-N2-C9	123.1(4)

Symmetry codes: a: 1–*x*, 1–*y*, 1–*z* for complex **2**



Hydrogen atoms, except those forming hydrogen bonds, are omitted for clarity

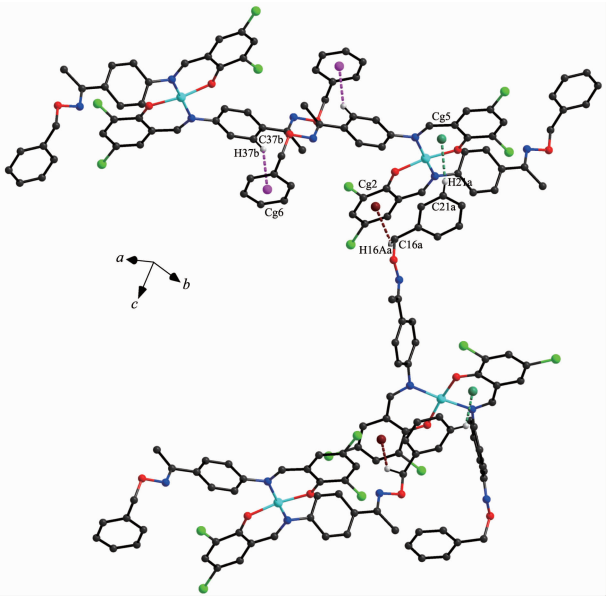
Fig.5 Intramolecular hydrogen bonds of complex **1**



Hydrogen atoms are omitted for clarity; Symmetry codes: a: 1/2−*x*, 3/2−*y*, 2−*z*; b: *x*, 2−*y*, 1/2+*z*; c: −*x*, *y*, 3/2−*z*

Fig.6 View of 3D supramolecular structure of complex **1** showing the formation of four $\pi\cdots\pi$ stacking interaction

layer supramolecular structure. This linkage is further stabilized by three C−H$\cdots\pi$ stacking interactions (Fig. 7, Table 6). Thus, the crystal packing of complex **1** shows that a 3D supramolecular network structure form through intermolecular C−H$\cdots\pi$ hydrogen bonding and $\pi\cdots\pi$ stacking interactions (Fig.8). Consequently, the intermolecular non-classical hydrogen-bonding plays a very important role in the construction of supramolecular networks structure.



Hydrogen atoms, except those forming hydrogen bonds, are omitted for clarity; Symmetry code: a: *x*, 2−*y*, −1/2+*z*; b: 1/2−*x*, 1/2−*y*, 2−*z*

Fig.7 View of the C−H$\cdots\pi$ stacking interaction of complex **1**

Table 4 Putative Hydrogen-bonding for complexes 1 and 2

D−H···A	*d*(D-H) / nm	*d*(H···A) / nm	*d*(D···A) / nm	∠DHA / (°)
Complex **1**				
C8−H8C···O2	0.096	0.216	0.261 7(6)	108
C30−H30A···O4	0.096	0.228	0.269 7(6)	105
C33−H33···N4	0.093	0.240	0.271 1(6)	100
C40−H40···N4	0.093	0.262	0.304 2(6)	108
Complex **2**				
C1−H1C···O1	0.096	0.223	0.264 0(8)	105

Table 5 Putative $\pi\cdots\pi$ stacking interactions for complex 1

Ring(I)	Ring(J)	*d*(Cg···Cg) / nm	*d*(Cg(I)···perp) / nm	*d*(Cg(J)···perp) / nm	Slippage
Cg1a	Cg2	0.374 3(2)	0.328 72(14)	0.329 61(17)	0.177 3
Cg1	Cg3b	0.359 1(3)	0.344 75(14)	0.351 8(2)	0.072 1
Cg2	Cg2a	0.366 8(2)	0.330 40(17)	0.330 38(17)	0.159 3
Cg4	Cg4c	0.365 5(3)	0.361 97(18)	0.361 96(18)	0.050 6

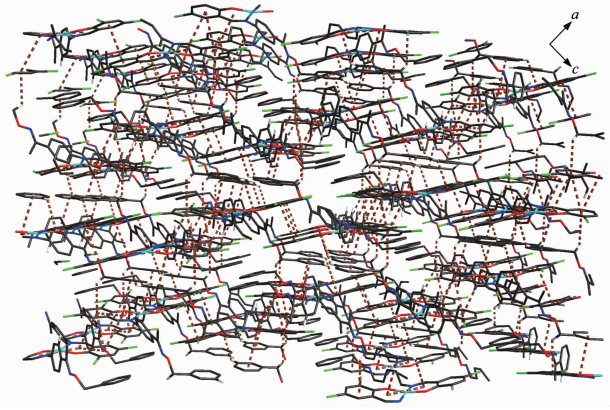
Cg1, Cg2, Cg3, Cg4 are the centroids of ring Cu1-O1-C3--C2-C1-N1, C2-C7, C17-C22, C24-C29 of complex **1**, respectively; Symmetry codes: a: 1/2−*x*, 3/2−*y*, 2−*z*; b: *x*, 2−*y*, 1/2+*z*; c: −*x*, *y*, 3/2−*z*

Table 6　Putative C–H···π stacking interactions for complex 1

D–H···A	*d*(D-H) / nm	*d*(H···A) / nm	*d*(D···A) / nm	∠DHA / (°)
Complex **1**				
C16a–H16Aa···Cg2	0.097	0.295	0.352 3(5)	119
C21a–H21a···Cg5	0.093	0.278	0.333 2(7)	119
C37b–H37b···Cg6	0.093	0.297	0.379 5(5)	148
Complex **2**				
C15–H15···Cg7a	0.082	0.290	0.365 1(6)	139
C22–H22···Cg8a	0.093	0.291	0.366 3(7)	139

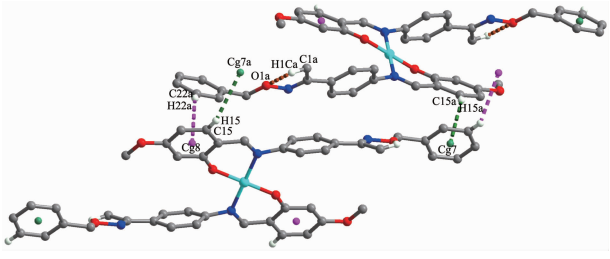
Cg2, Cg5, Cg6 are the centroids of ring C2~C7, Cu1-O3-C25-C24-C23-N3, C39~C44 of complex **1**, respectively; Cg7 and Cg8 are the centroids of benzene ring C18~C23, C10~C15 of complex **2**, respectively; Symmetry codes: a: *x*, 2–*y*, –1/2+*z*; b: 1/2–*x*, 1/2–*y*, 2–*z* for **1**; 1–*x*, 1/2+*y*, 1/2–*z* for **2**.

In complex **2**, the crystal structure of complex **2** is stabilized by pair of intramolecular C1–H1C···O1 hydrogen bonds generating five-membered ring motifs (Fig.9, Table 4). This linkage is further stabilized by two pairs of C15–H15···π (Cg7) and C22–H22···π (Cg8) interactions to form the 2D channel supramolecular structure along the *b* axis (Fig.10, Table 6).



Hydrogen atoms, except those forming hydrogen bonds, are omitted for clarity

Fig.8　View of the 3D supramolecular structure of complex **1**



Hydrogen atoms, except those forming hydrogen bonds, are omitted for clarity; Symmetry codes: a: 1–*x*, 1/2+*y*, 1/2–*z*

Fig.9　Putative intramolecular hydrogen bonds and intermolecular C–H···π stacking interaction of complex **2**

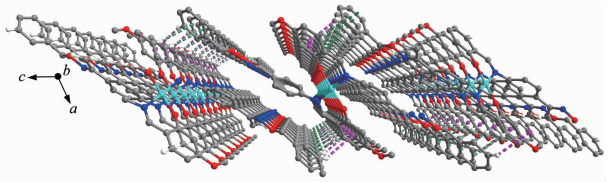


Fig.10　View of the 2D channel supramolecular structure within complex **2** along the *b* axis

3　Conclusions

Two oxime-type Schiff base mononuclear Cu(Ⅱ)/Ni(Ⅱ) complexes **1** and **2** have been synthesized and characterized structurally. The complexes **1** and **2** are all tetra-coordinated by two nitrogen atoms and two oxygen atoms of two deprotonated L^- units defining the N_2O_2 basal plane. The coordination environment around center metal ion is regarded as the distorted square-planar geometry. Complexes **1** and **2** also form a 3D and 2D supramolecular structures via different intermolecular interaction C–H···π and π···π stacking interactions, respectively. Consequently, the intermolecular non-classical hydrogen-bonding plays a very important role in the construction of supramolecular networks structure.

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