呋喃取代三芳基三唑的单核铜配合物中共存两个不同的铜离子

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摘要:分别以3-(2-吡啶基)-4-苯基-5-(2-呋喃基)-1,2,4-三氮唑(L¹)和3-(2-吡啶基)-4-(对氟苯基)-5-(2-呋喃基)-1,2,4-三氮唑(L²)作为配体,合成了2个新的单核铜配合物:*trans*-[Cu(L¹)₂(NO₃)₂]_{0.5}[Cu(L¹)₂(H₂O)₂]_{0.5}(NO₃)(1)和*trans*-[Cu(L²)₂(NO₃)₂]_{0.5}[Cu(L²)₂(H₂O)₂]_{0.5}(NO₃)(1)和*trans*-[Cu(L²)₂(NO₃)₂]_{0.5}[Cu(L²)₂(H₂O)₂]_{0.5}(NO₃)(1)和*trans*-[Cu(L²)₂(NO₃)₂]_{0.5}[Cu(L²)₂(H₂O)₂]_{0.5}(NO₃)·2CH₃OH(2)。对它们进行了红外、元素分析、热重分析、单晶结构和粉末X射线衍射表征。2个配合物都属于三斜晶系, P¹空间群。单晶结构表明,配合物1和2中均有2个不同的铜离子,且都处于一个扭曲的八面体[CuN₄O₂]配位环境,但在轴向上一个铜离子与2个水分子配位,另一个铜离子则与2个硝酸根配位。配体的吡啶氮原子和三氮唑的一个氮原子参与配位,而呋喃的氧原子不配位。配合物晶体中存在C—H…N、C—H…O、O—H…O氢键和C—H…π相互作用,从而连接单核配合物形成二维层状结构。

关键词:呋喃;铜配合物;晶体结构;三芳基三氮唑
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Coexistence of Two Unique Cu(II) Ions in Mononuclear Cu(II) Complexes with Furanyl Substituted Triaryltriazoles

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Abstract: Two new mononuclear Cu(II) complexes, *trans*-[Cu(L¹)₂(NO₃)₂]_{0.5}[Cu(L¹)₂(H₂O)₂]_{0.5}(NO₃) (1) and *trans*-[Cu(L²)₂ (NO₃)₂]_{0.5}[Cu(L²)₂(H₂O)₂]_{0.5}(NO₃) · 2CH₃OH (2) (L¹=3-(2-pyridyl)-4-phenyl-5-(2-furanyl)-1,2,4-triazole, L²=3-(2-pyridyl) -4-(*p*-fluorophenyl)-5-(2-furanyl)-1,2,4-triazole), were synthesized and characterized by FT-IR, elemental analyses, thermogravimetric analyses, single-crystal X-ray crystallography and powder X-ray diffraction. Both complexes 1 and 2 crystallize in triclinic system with space group *P*1. The crystal structure analysis shows that there are two crystallographically different Cu(II) ions in 1 and 2 and each Cu(II) ion lies in a distorted octahedral [CuN₄O₂] environment. However, in the axial position one Cu(II) ion is coordinated with two water molecules, while another Cu(II) ion with two nitrate ions. The L¹ or L² ligand coordinates with Cu(II) via one pyridine N atom and one triazole N atom, while the furanyl group does not participate in coordination. In 1 and 2 there are some intermolecular C—H···N, C—H···O, O—H···O hydrogen bonding and C—H···*π* interactions, linking the mononuclear complex (1 or 2) to form a two-dimensional network. CCDC: 2053558, 1; 2053559, 2.

Keywords: furan; Cu(II) complex; crystal structure; triaryltriazole

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0 Introduction

1,2,4-triazole derivatives as a kind of important nitrogen-rich heterocyclic compounds have been widely applied in biological science, coordination chemistry, material chemistry, medicinal chemistry and organic synthetic chemistry because of their unique structures and chemical properties^[1-4]. Especially, 3,4,5- triarylsubstituted 1,2,4-triazoles have received extensive attention in coordination chemistry owing to their versatile coordination modes^[5-7]. It is worthwhile to note that some Cu(II) complexes based on triaryltriazoles can exhibit interesting antimicrobial and anticancer properties^[8-9]. Recently, a series of Cu(II) complexes based on triaryltriazoles with pyridyl, quinolyl, thienyl groups have been reported by our group^[10-16]. However, 2-furanyl substituted triaryltriazoles have been less explored till now^[17-19]. As a continued research on the triaryltriazoles, we designed and synthesized two new 2-furanyl substituted triaryltriazoles: 3-(2-pyridyl)-4-phenyl-5-(2furanyl) - 1, 2, 4 - triazole (L¹) and 3 - (2 - pyridyl) - 4 - (p fluorophenyl)-5-(2-furanyl)-1,2,4-triazole (L²) (Scheme 1). Herein, we report the syntheses, crystal structures and thermogravimetric analyses of two mononuclear Cu(II)complexes with the synthesized ligands: $trans - [Cu(L^1)_2(NO_3)_2]_{0.5} [Cu(L^1)_2(H_2O_2)_2]_{0.5} (NO_3)$ (1) and $trans - [Cu(L^2)_2(NO_3)_2]_{0.5}[Cu(L^2)_2(H_2O)_2]_{0.5}(NO_3) \cdot 2CH_3OH$ (2). Notably, it is first observed that two distinct Cu(II) ions coexist in these mononuclear Cu (II) complexes with furanyl substituted triaryltriazoles^[20].



Scheme 1 Structures of ligands L1 and L2

1 Experimental

1.1 Materials and measurements

All chemicals used were of analytical grade. Solvents were purified by conventional methods. The ligands L¹ and L² were synthesized via a similar literature method^[21]. Elemental analyses (C, H, N) were carried out with a Thermo Finnigan Flash 1112A elemental analyzer. IR spectra were recorded on a Nicolet Avatar 380 FT - IR instrument with KBr pellets in a range of 4 000~400 cm⁻¹. Thermogravimetric analysis (TGA) was performed with a simultaneous NETZSCH STA 449C thermal analyzer under flowing nitrogen from 25 to 800 °C at a heating rate of 5 °C ·min⁻¹. Powder X - ray diffraction (PXRD) data were collected on a Bruker D8 ADVANCE diffractometer using Cu K α radiation (λ =0.154 06 nm) at 40 kV and 40 mA in a range of 5°~50°.

1.2 Syntheses of complexes 1 and 2

trans-[Cu(L¹)₂(NO₃)₂]_{0.5}[Cu(L¹)₂(H₂O)₂]_{0.5}(NO₃) (1): A solution of Cu(NO₃)₂·3H₂O (0.125 mmol) in water (1 mL) was added to a solution of L¹ (0.25 mmol) in MeOH (1 mL). The mixture was stirred for 4 h at room temperature. A resulting green product was filtered and washed with H₂O, then dried under vacuum to give 0.116 mmol (92.8%) of complex **1**. The well-shaped single crystals of **1** suitable for X-ray diffraction were obtained by slow evaporation from the MeOH solution of the complex. Elemental analysis Calcd. for C₃₄H₂₆CuN₁₀O₉(%): C, 52.21; H, 3.35; N, 17.91. Found (%): C, 52.46; H, 3.17; N, 17.76. FT-IR (KBr, cm⁻¹): 3 479(w), 3 058(m), 3 011(w), 1 615(m), 1 504(s), 1 384 (vs), 1 308(s), 1 021(m), 751(m), 695(m).

trans-[Cu(L^2)₂(NO₃)₂]_{0.5} [Cu(L^2)₂(H₂O)₂]_{0.5}(NO₃) · 2CH₃OH (**2**): The prepared procedure was the same as that for **1** except using L² (0.25 mmol) to replace L¹. Yield: 87.9%. Elemental analyses Calcd. for C₃₆H₃₂CuF₂N₁₀O₁₁(%): C, 49.01; H, 3.66; N, 15.88. Found(%): C, 49.33; H, 3.45; N, 16.09. FT-IR (KBr, cm⁻¹): 3 351(w), 2 923(w), 1 613(m), 1 515(vs), 1 384 (s), 1 149(m), 836(m), 743(m).

1.3 Crystal structure determination

The well-shaped single crystals of **1** and **2** were selected for X-ray diffraction study. The unit cell parameters and intensity data were collected at 296(2) K on a Bruker SMART APEX II CCD diffractometer using a graphite-monochromated Mo $K\alpha$ (λ =0.071 073 nm) radiation. The structures were solved by direct methods and refined on F^2 by full-matrix least squares procedures using SHELXTL software^[22]. All nonhydrogen atoms were refined anisotropically. All hydrogen atoms were fixed in calculated positions and refined isotropically. The coordinated NO₃⁻ ions in **2** were disordered over two positions with an occupancy of 0.698(13) for N9, O3, O4 and O5 and 0.302(13) for N9A, O3A, O4A and O5A. The crystallographic data of **1** and **2** are listed in Table 1 and the selected bond lengths and angles are provided in Table 2.

CCDC: 2053558, 1; 2053559, 2.

Complex	1	2
Empirical formula	$C_{34}H_{26}CuN_{10}O_9$	$C_{36}H_{32}CuF_2N_{10}O_{11}$
Formula weight	782.19	882.25
Crystal system	Triclinic	Triclinic
Space group	$P\overline{1}$	$P\overline{1}$
<i>a /</i> nm	0.871 20(11)	0.869 34(13)
<i>b</i> / nm	1.363 75(17)	1.586 6(2)
<i>c</i> / nm	1.531 24(19)	1.599 9(2)
α / (°)	85.602(2)	103.974(2)
β/(°)	79.815(2)	104.405(2)
γ / (°)	72.386(2)	100.992(2)
V / nm^3	1.706 1(4)	1.998 9(5)
Z	2	2
$D_{\rm c} / ({ m g} {}^{m \cdot} { m cm}^{-3})$	1.523	1.466
μ / $ m mm^{-1}$	0.712	0.627
F(000)	802	906
Crystal size / mm	0.20×0.11×0.10	0.19×0.17×0.11
θ range / (°)	1.57~25.00	1.37~25.00
Reflection collected	12 406	14 471
Independent reflection	5 963 (R_{int} =0.026 0)	6 989 (<i>R</i> _{int} =0.022 1)
Reflection observed $[I\!\!>\!\!2\sigma(I)]$	4 656	5 980
Data, restraint, parameter	5 963, 6, 490	6 989, 36, 585
Goodness-of-fit on F^2	1.078	1.082
$R_1, wR_2 [I > 2\sigma(I)]$	0.049 4, 0.136 6	0.048 9, 0.131 1
R_1, wR_2 (all data)	0.064 0, 0.145 5	0.056 6, 0.136 1
$(\Delta \rho)_{\rm max}, (\Delta \rho)_{\rm min} / (e \cdot nm^{-3})$	818, -435	458, -752

 Table 1
 Crystal data and structure refinements for 1 and 2

Та	ble	e 2	Se	lected	bond	lengths	(nm)) and	angle	es (°)) for	comp	lexes	1 and	2
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		1			
Cu1—N1	0.203 3(2)	Cu1—N2	0.199 6(2)	Cu1—03	0.244 4(3)
N2—N3	0.137 4(3)	N4—C7	0.136 8(4)	N9—03	0.126 2(4)
Cu2—N5	0.206 0(3)	Cu2—N6	0.199 0(3)	Cu2—O1W	0.234 6(3)
N6—N7	0.137 6(4)	N8—C24	0.136 9(4)		
N1—Cu1—N2	80.56(10)	N1—Cu1—O3	92.00(13)	N2-Cu1-03	95.44(12)
C1—N1—C5	117.8(3)	C6—N4—C7	105.1(2)	C8-01-C11	105.5(3)
N5—Cu2—N6	79.55(11)	N5—Cu2—O1W	90.56(13)	N6—Cu2—O1W	90.45(12)
C18—N5—C22	118.2(3)	C23—N8—C24	104.4(3)	C25—O2—C28	105.5(3)

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Continued Table 2										
				2	2					
Cu1—N1	0.203 0(2)		Cu1—	N2		0.199 7(2	2)	Cu1—03	0.245 3(2)	
N2—N3	0.137 6(3)		N4—0	27		0.137 5(3	i)	C15—F1	0.153 3(5)	
Cu2—N5	0.206 5(2)		Cu2—	N6		0.197 7(2	2)	Cu2—O1W	0.235 7(3)	
N6—N7	0.137 2(3)		N8—C	224		0.137 6(4	.)	C32—F2	0.152 9(4)	
N1—Cu1—N2	80.09(9)	Ν	N1—Cu1	-03		87.65(12	2)	N2—Cu1—O3	92.28(13)	
C1—N1—C5	118.4(2)	(26—N4-	—С7		104.7(2)		C8-01-C11	105.4(3)	
N5—Cu2—N6	79.75(9)	Ν	N5—Cu2	-01W		87.31(11	l)	N6—Cu2—O1W	88.56(10)	
C18—N5—C22	118.5(3)	(223—N8	-C24		104.7(2)		C25—O2—C28	105.8(3)	

2 Results and discussion

2.1 Synthesis

Asymmetrically 2-furanyl substituted 3,4,5-triaryl-1, 2, 4 - triazoles (L¹ and L²) reacting with Cu(NO₃)₂· 3H₂O in molar ratio of 2:1 produced two mononuclear complexes, *trans*-[Cu(L¹)₂(NO₃)₂]_{0.5}[Cu(L¹)₂(H₂O)₂]_{0.5}(NO₃) (1) and *trans*-[Cu(L²)₂(NO₃)₂]_{0.5}[Cu(L²)₂(H₂O)₂]_{0.5}(NO₃) · 2CH₃OH (2), which were stable in air. Yields for 1 and 2 were 92.8% and 87.9%, respectively. The elemental analyses were satisfactory and reveal that both 1 and 2 contain one Cu(II) ion, two triazole ligands (L¹ for 1 and L² for 2), two NO₃⁻ ions and one water molecule, except that 2 still has two methanol molecules.

2.2 Crystal structures of 1 and 2

The projection of structures of **1** and **2** is shown in Fig. 1 together with the atomic labeling system. Both **1**

and **2** crystallize in the triclinic space group *P*1 and an inversion center is found in the Cu(II) ion. Because 1 and 2 have a similar structure, herein, only the structure of **1** is described detailedly. The asymmetric unit of 1 contains two crystallographically different Cu (II) ions (the occupancy factors of both Cu1 and Cu2 are 0.5), two L^1 ligands, two NO_3^- ions and one H_2O molecule, which is in consistent with the elemental analysis result. Both Cu1 and Cu2 ions lie in a distorted octahedral [CuN4O2] environment with four N atoms from two L^1 ligands in the equatorial plane. However, in the axial position the Cu1 ion is coordinated by two nitrate ions, while the Cu2 ion is coordinated by two water molecules. Each L^1 ligand coordinates to Cu1 (or Cu2) via N1 (or N5) atom of the pyridyl and N2 (or N6) atom of the triazole, while the 2-furanyl group does not par-



All H atoms and disordered atoms are omitted for clarity; Symmetry codes: ⁱ 1–x, 2–y, 1–z; ⁱⁱ –x, 1–y, 1–z, ⁱⁱⁱ 1–x, 1–y, 1–z, ⁱⁱⁱ 1–x, 2–y, 2–z; ⁱⁱ –x, 1–y, 2–z; ⁱⁱ –x, 1–z; 2–x, 2–x, 2–x; 2–x

Fig.1 Projection of structures of 1 and 2 with 10% thermal ellipsoids probability

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ticipate in coordination. It is worthwhile to note that two distinct Cu(II) ions are observed to coexist in present mononuclear Cu (II) complexes for the first time, which is quite different from the mononuclear Cu(II) complexes with the quinolyl or thienyl substituted triarvltriazoles^[15-16]. The distance of Cu1-O3 (0.244 4 nm) is longer than the Cu2-O1W one (0.234 6 nm) (Table 2). The Cu-N bond lengths are within the normal ranges found for the octahedral Cu(II) complexes^[10-16]. The ligand L^1 in **1** is non-planar. The triazole coordinating to Cu1 makes dihedral angles of 47.5(2)°, 8.4(2)° and $82.1(2)^{\circ}$ with the 2-furanyl ring, the pyridyl ring and phenyl ring, respectively, while the triazole coordinating to Cu2 makes dihedral angles of $5.5(2)^{\circ}$, $5.3(2)^{\circ}$ and 88.9(2)° with the 2-furanyl ring, the pyridyl ring and phenyl ring, respectively (Table 3). The corresponding dihedral angles in L^2 of **2** are also given in Table 3.

There are rich intermolecular hydrogen bonds and C—H··· π interactions in **1** (Fig. S1, Table S1 in Supporting information), associated with the closer crystal packing. These hydrogen-bond interactions include: (1) between pyridyl and triazole rings (C1—H1A····N3ⁱ and C18—H18A····N7ⁱⁱⁱ); (2) between pyridyl rings and

Table 3 Dihedral angles (°) for 1 and 2 $\,$

Complex	Py ^a to Trz	Ph ^b to Trz	$Fu^{\rm c}$ to Trz
1 (Cu1)	8.4(2)	82.1(2)	47.5(2)
1 (Cu2)	5.3(2)	88.9(2)	5.5(2)
2 (Cu1)	4.3(2)	88.4(2)	17.9(2)
2 (Cu2)	8.3(2)	87.0(2)	2.0(3)

^a Py: pyridyl ring, Trz: 1,2,4-triazole; ^b Ph: phenyl ring; ^c Fu: furanyl ring

NO₃⁻ anions (C2-H2A···O5ⁱⁱ, C3-H3A···O3ⁱⁱ and C20—H20A····O8); (3) between furan group and NO_3^{-1} anion (C9—H9A····O8); (4) between phenyl and NO_3^{-1} anions (C13-H13A····O5ⁱ, C17-H17A····O6, C30-H30A····O4^{iv} and C34—H34A····O7ⁱⁱ); (5) between water molecule and NO3⁻ anions (O1W-H1WA...O7ⁱⁱ and O1W-H1WB····O3ⁱⁱ). In addition, there are an intermolecular edge-to-face C—H $\cdots \pi$ interaction (C11— H11A... π (Ph)^v) and three intramolecular edge-to-face C—H··· π interactions (C4—H4A··· π (Ph), C21—H21A $\cdots \pi(Ph)$ and C26—H26A $\cdots \pi(Ph)$). These extensive hydrogen-bond and C—H $\cdots \pi$ interactions connect the mononuclear units, NO3⁻ anions and lattice water molecules into a 2D layer (Fig.2). The corresponding hydrogen-bonding and C—H··· π interactions of **2** are also shown in Fig.S1 and Table S1.



Fig.2 Two-dimensional networks of 1 and 2

2.3 IR spectra

In the IR spectra of 1 and 2, a weak broad peak at 3 479 cm⁻¹ (1) or 3 370 cm⁻¹ (2) can be attributed to the O—H stretching vibrations of water molecules. A medium band at 1 615, 1 504 cm⁻¹ (1) or 1 613, 1 515 cm⁻¹ (2) can be assigned to the coordinated pyridyl ring vibration^[11]. A strong band at 1 384 cm⁻¹ is assigned to characteristic N=O stretching vibrations of NO₃⁻ in 1 and $2^{[15]}$. In addition, the stretching vibration of F—C (Ph) in 2 can be observed at 1 149 cm^{-1[23]}. These features are in consistent with the results of the X - ray analyses.

2.4 Thermal stability and PXRD

As shown in Fig. 3, the first weight loss of 2.3% between 25 and 178 °C for **1** was observed due to the loss of one coordinated water molecule (Calcd. 2.3%). Then **1** started to decompose. The remaining weight of 10.0% after heating to 500 °C is owing to the final residue of CuO (Calcd. 10.2%).

The first weight loss of 9.0% between 25 and



Fig.3 TGA curves of 1 and 2

268 °C in **2** was found because of the loss of one coordinated water molecule and two lattice methanol molecules (Calcd. 9.3%). Then **2** began to collapse. The remaining weight of 10.8% after heating to 500 °C is due to the final residue of CuO, in agreement with the calculated value of 9.2%.

The PXRD patterns and simulated ones of **1** and **2** are shown in Fig.S2. The experimental patterns were in well consistent with the simulated ones from the single X-ray crystal data, indicating the high phase purity of the bulk products of **1** and **2**.

3 Conclusions

Two new mononuclear Cu(II) complexes based on the 2-furanyl substituted triaryltriazoles have been synthesized and structurally characterized by FT-IR and X-ray crystallography. It is first observed that two distinct Cu(II) ions coexist in these mononuclear Cu(II) complexes with furanyl substituted triaryltriazoles.

Supporting information is available at http://www.wjhxxb.cn

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