

一个双核镍配合物 $[\text{Ni}_2(\text{Htda})_2(\text{H}_2\text{O})_6]\cdot 4\text{H}_2\text{O}$ 的合成、晶体结构和性质

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摘要: 双核金属配合物对于生物化学和磁化学都是非常重要的。在本文中, 1,2,3-三氮唑-4,5-二羧酸(H_3tda)配体和硝酸镍在常温常压下反应得到了 1 个三氮唑桥联的双核镍配合物 $[\text{Ni}_2(\text{Htda})_2(\text{H}_2\text{O})_6]\cdot 4\text{H}_2\text{O}$ (**1**), 并通过元素分析、红外光谱、X-射线单晶衍射、热重分析以及超导量子干涉仪(SQUID)对其晶体结构和磁、热性质进行了表征。晶体数据表明该配合物属三斜晶系, 空间群为 $P\bar{1}$ 。在配合物 **1** 中, 2 个 1,2,3-三氮唑-4,5-二羧酸配体利用 2 个相邻的氮原子桥联 2 个金属镍离子形成一个双桥的双核镍配合物。这些双核镍单元又通过体系中存在的大量的氢键相互作用形成了三维超分子骨架。磁性分析显示双核镍单元内镍镍之间存在反铁磁相互作用。

关键词: 双核镍配合物; 1,2,3-三氮唑-4,5-二羧酸; 晶体结构; 氢键; 磁性

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A Dinuclear Ni(II) Complex $[\text{Ni}_2(\text{Htda})_2(\text{H}_2\text{O})_6]\cdot 4\text{H}_2\text{O}$: Synthesis, Crystal Structure and Properties

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Abstract: The dinuclear metal complexes are important not only for biochemistry but also for magnetochemistry. In this paper, a dinuclear Ni(II) complex $[\text{Ni}_2(\text{Htda})_2(\text{H}_2\text{O})_6]\cdot 4\text{H}_2\text{O}$ (**1**) with 1,2,3-triazole-4,5-dicarboxylic acid (H_3tda) as ligands has been synthesized and characterized by element analysis, IR spectra, single-crystal X-ray diffraction, thermo gravimetric analysis and superconducting quantum interference device (SQUID). The crystallographic data shows that complex **1** crystallizes in triclinic space group $P\bar{1}$ with $a=0.721\ 19(16)$ nm, $b=0.812\ 81(18)$ nm, $c=0.940\ 2(2)$ nm, $\alpha=109.526(4)^\circ$, $\beta=91.868(4)^\circ$, $\gamma=103.304(4)^\circ$, $V=0.501\ 88(19)$ nm³, $\text{C}_8\text{H}_{22}\text{N}_6\text{Ni}_2\text{O}_{18}$, $M_r=607.74$, $D_c=2.011\ \text{g}\cdot\text{cm}^{-3}$, $\mu(\text{Mo } K\alpha)=1.982\ \text{mm}^{-1}$, $F(000)=312$, $\text{GOF}=1.037$, $Z=1$, the final $R_1=0.045\ 1$ and $wR_2=0.117\ 7$ for $I>2\sigma(I)$. In **1**, two 1,2,3-triazole-4,5-dicarboxylic acid ligands bridge two Ni(II) ions by two neighboring nitrogen atoms in triazole ring to form a dinuclear Ni(II) unit, which is further connected with each others through plenty of hydrogen-bond interactions to construct a three-dimensional (3D) supramolecular architecture. The magnetic study shows that the antiferromagnetic interactions exist between Ni(II) ions in dinuclear Ni(II) units. CCDC: 747631.

Key words: dinuclear Ni(II) complex; 1,2,3-triazole-4,5-dicarboxylic acid; crystal structure; hydrogen bond; magnetic property

Since a lot of metalloenzymes, such as leucine aminopeptidase, urease, methionineaminopeptidase, arginase, P1 nuclease, DNA polymerase I, phospholi-

pase C, and alkaline phosphatase, contain a dinuclear metal active site featuring Zn(II), Ni(II), Co(II), Co(III), Cu(II) and Mn(II), etc. bridged by carboxylate or

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hydroxide ion, artificial models for these dinuclear metalloenzymes are of particular interest because of their ability to increase the understanding of the catalytic mechanism of the enzymes and the possible applications in biotechnology^[1-7]. Consequently, the dinuclear metal complexes with two metal ions bound by the bridging ligands have attracted a great deal of attentions^[8]. Similarly, these dinuclear metal compounds are also useful for development of magnetic models and investigation of the magnetic properties of multimetal compounds.

1,2,3-triazole-4,5-dicarboxylic acid (H_3tda) is a multifunctional ligand with abundant coordination modes and multiple coordination sites involving three triazole nitrogen atoms and four carboxylate oxygen atoms and can react with various metals to produce intriguing poly-dimensional structures with complicated topologies and/or useful properties^[9-11]. Furthermore, it has three abstractable hydrogens, which is beneficial to meet the charge-balance requirement of the system. In this paper, we used H_3tda as ligands reacting with $Ni(NO_3)_2 \cdot 6H_2O$ and obtained a triazolato-bridged dinuclear $Ni(II)$ complex $[Ni_2(Htda)_2(H_2O)_6] \cdot 4H_2O$ (**1**), and its synthesis, crystal structure, magnetic and thermal properties are reported herein.

1 Experimental

1.1 General

All chemicals are reagent grade and used as received. H_3tda was synthesized according to the literature methods^[12]. Elemental analyses for C, H and N were performed on a Perkin-Elmer 240C analyzer. Infrared spectra were recorded on a Vector22 Bruker Spectrophotometer with KBr pellets in the 400~4 000 cm^{-1} regions. Magnetic susceptibility measurements of polycrystalline samples were measured over the temperature range 1.8~300 K with a Quantum Design MPMS-XL7 SQUID magnetometer. Thermogravimetric

analyses were collected on a Perkin-Elmer Pyris 1 TGA analyzer from room temperature to 750 $^{\circ}C$ with a heating rate of 20 $^{\circ}C \cdot min^{-1}$ under nitrogen.

1.2 Synthesis of $[Ni_2(Htda)_2(H_2O)_6] \cdot 4H_2O$ (**1**)

H_3tda (0.1 mmol, 15.7 mg) dissolved in 10 mL of deionized water was slowly dropped into $Ni(NO_3)_2 \cdot 6H_2O$ (0.1 mmol, 29.1 mg) dissolved in 5 mL of deionized water, with continuous stirring at room temperature for 1 h. The resulting green solutions were filtered and left to stand. Green block crystals of **1** suitable for X-ray diffraction were obtained by slow evaporation of the solvents for about 10 days. Yield: 62%. Anal. Calcd for $C_8H_{22}N_6Ni_2O_{18}$: C, 15.81; H, 3.65; N, 13.83. Found: C, 15.94; H, 3.58; N, 13.74%. IR (KBr)/ cm^{-1} : 3 376 vs, 1 720 vs, 1 554 s, 1 376 s, 1 228 m, 781 m, 622 w.

1.3 Structure determination

A single crystal of **1** with dimensions of 0.12mm \times 0.15mm \times 0.17mm was mounted on a Bruker Smart Apex CCD diffractometer equipped with a graphite-monochromatic Mo $K\alpha$ radiation ($\lambda = 0.071\ 073\ nm$) radiation using the φ - ω scan mode in the range $2.31^{\circ} \leq \theta \leq 24.99^{\circ}$ at 291(2) K. A total of 2 502 reflections were collected and 1 742 were independent with $R_{int} = 0.043\ 3$, of which 1 576 were observed with $I > 2\sigma(I)$. Raw frame data were integrated with the SAINT program^[13]. The structure was solved by direct methods and refined by full-matrix least-squares on F^2 using SHELX-97^[14]. An empirical absorption correction was applied with the program SADABS^[15]. All non-hydrogen atoms were refined anisotropically. Hydrogen atom H(3) was located in the difference Fourier maps and all other hydrogen atoms were set in calculated positions and refined by a riding mode, with a common thermal parameter. Crystallographic details have been summarized in Table 1. Selected bond lengths and angles and hydrogen-bonding geometry parameters for **1** are listed in Tables 2 and 3.

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Table 1 Crystallographic data for complex **1**

Empirical formula	$C_8H_{22}N_6Ni_2O_{18}$	V / nm^3	0.501 88(19)
Formula weight	607.74	Z	1
Crystal size / mm	0.12 \times 0.15 \times 0.17	$D_c / (g \cdot cm^{-3})$	2.011
Temperature / K	291(2)	μ / mm^{-1}	1.982

Continued Table 1

Crystal system	Triclinic	$F(000)$	312
Space group	$P\bar{1}$	θ range / (°)	2.31~24.99
a / nm	0.721 19(16)	Reflections collected / unique	2 502 / 1742
b / nm	0.812 81(18)	Goodness of fit on F^2	1.037
c / nm	0.940 2(2)	R_1 [$I > 2\sigma(I)$]	0.045 1
α / (°)	109.526(4)	wR_2 [$I > 2\sigma(I)$]	0.117 7 ^a
β / (°)	91.868(4)	Parameters	154
γ / (°)	103.304(4)	$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ / ($\text{e}\cdot\text{nm}^{-3}$)	933, -591

^a $w=1/[\sigma^2(F_o^2)+(0.072\ 1P)^2+0.325\ 9P]$ where $P=(F_o^2+2F_c^2)/3$.

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

Ni(1)-N(1) ⁱ	0.204 9(3)	Ni(1)-N(2)	0.203 7(3)	Ni(1)-O(1) ⁱ	0.210 7(3)
Ni(1)-O(5)	0.208 8(3)	Ni(1)-O(6)	0.210 2(3)	Ni(1)-O(7)	0.205 2(3)
N(2)-Ni(1)-N(1) ⁱ	96.83(13)	N(2)-Ni(1)-O(7)	89.41(13)	N(1)i-Ni(1)-O(7)	173.75(12)
N(2)-Ni(1)-O(5)	91.65(13)	N(1)i-Ni(1)-O(5)	91.21(13)	O(7)-Ni(1)-O(5)	89.04(13)
N(2)-Ni(1)-O(1) ⁱ	176.59(12)	N(1)i-Ni(1)-O(1) ⁱ	79.78(12)	O(7)-Ni(1)-O(1) ⁱ	93.99(11)
O(5)-Ni(1)-O(1) ⁱ	88.09(12)	N(2)-Ni(1)-O(6)	93.15(13)	N(1)i-Ni(1)-O(6)	89.84(13)
O(7)-Ni(1)-O(6)	89.38(13)	O(5)-Ni(1)-O(6)	174.93(11)	O(1)i-Ni(1)-O(6)	87.22(12)

Symmetry code: ⁱ $-x, -y, -z$.

Table 3 Hydrogen-bonding geometry parameters for complex 1

D-H...A	$d(\text{D-H})$ / nm	$d(\text{H}\cdots\text{A})$ / nm	$d(\text{D}\cdots\text{A})$ / nm	$\angle(\text{DHA})$ / (°)
O(3)-H(3)···O(2)	0.079(6)	0.173(6)	0.251 0(4)	168(6)
O(5)-H(5A)···O(2) ⁱⁱ	0.085	0.195	0.270 5(4)	146.8
O(5)-H(5B)···O(9) ⁱ	0.085	0.202	0.278 0(5)	147.9
O(6)-H(6A)···O(8) ⁱⁱⁱ	0.085	0.214	0.283 1(5)	137.7
O(6)-H(6B)···O(9) ⁱⁱⁱ	0.085	0.210	0.271 7(5)	128.7
O(6)-H(6B)···O(6) ⁱⁱⁱ	0.085	0.250	0.279 0(6)	101.0
O(7)-H(7A)···O(4) ^{iv}	0.085	0.189	0.272 2(4)	167.4
O(7)-H(7B)···O(8) ^{iv}	0.085	0.209	0.291 2(5)	164.2
O(8)-H(8A)···O(5) ^v	0.085	0.209	0.289 5(5)	158.3
O(8)-H(8B)···O(7)	0.085	0.219	0.300 2(6)	159.7
O(9)-H(9B)···O(4) ^{vi}	0.085	0.197	0.273 1(4)	147.9
O(9)-H(9B)···O(3) ^{vi}	0.085	0.252	0.330 6(5)	154.8

Symmetry codes: ⁱ $-x, -y, -z$; ⁱⁱ $x, y, -1+z$; ⁱⁱⁱ $1-x, -y, -z$; ^{iv} $1-x, 1-y, -z$; ^v $1+x, y, z$; ^{vi} $1-x, 1-y, 1-z$.

2 Results and discussion

Strong absorption peak at $1\ 720\ \text{cm}^{-1}$ for $-\text{COOH}$ are observed, indicating that the carboxyl groups of H_3tda ligands are not all deprotonated^[16]. According to the difference Fourier maps, charge balance and the C-O bond lengths, we think that the O(3) atom is protonated.

2.1 Structure description

The X-ray crystallographic analysis reveals that **1**

is a dinuclear Ni (II) complex. An ORTEP view of complex **1** is shown in Fig.1. The asymmetric unit of **1** contains one nickel ion (Ni(1)), one Htda^{2-} ligand, three coordinated water molecules (O(5), O(6) and O(7)) and two lattice aqua molecules (O(8) and O(9)). The Htda^{2-} ligand acts in $\mu_3\text{-}\eta^2\text{N}, \text{O}, \eta^1\text{N}'$ fashion, chelating a Ni(1) center through a carboxylate oxygen atom (O(1)) and a triazole nitrogen atom (N(1)), and bridging another one crystallographically equivalent Ni(1) center through the

N(2) atom neighboring to the N(1) atom, respectively. Each Ni^{2+} ion is six-coordinated by a carboxylate oxygen atom and a triazole nitrogen atom from an Htda^{2-} ligand, a triazole nitrogen atom from another one Htda^{2-} ligand and three water molecules. The coordination geometry around Ni^{2+} ion is a slightly distorted octahedron. The two Ni^{2+} ions coordinated to one Htda^{2-} ligand are simultaneously bound to another one crystallographically equivalent Htda^{2-} ligand to form a dinuclear $\text{Ni}(\text{II})$ unit $[\text{Ni}_2(\text{Htda})_2]$. The $[\text{Ni}_2(\text{Htda})_2]$ unit is almost planar with the largest deviation of 0.005 48 nm for O(4) atom from the mean plane defined by the triazole ring. The $\text{Ni}(1) \cdots \text{Ni}(1)^i$ distance is 0.405 0(1) nm. The shortest $\text{Ni} \cdots \text{Ni}$ distance between the dinuclear $\text{Ni}(\text{II})$ units is 0.624 1(1) nm. The Ni-N bond lengths range from 0.203 7(3) to 0.204 9(3) nm and the Ni-O bond lengths range from 0.205 2(3) to 0.210 7(3) nm, which are all similar to those values found in other $\text{Ni}(\text{II})$ complexes^[17-23].

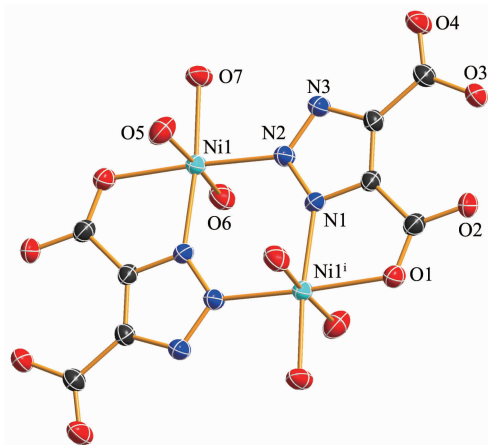
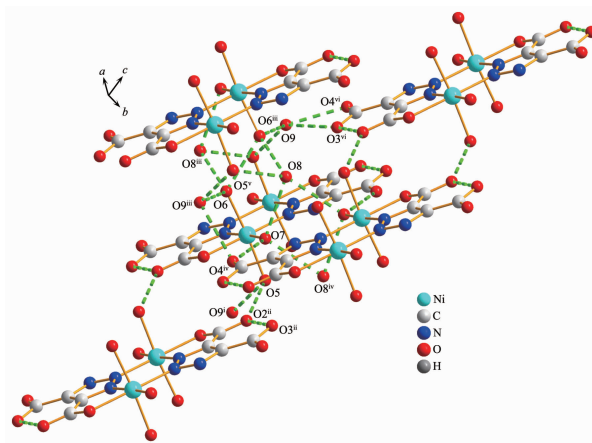


Fig.1 View of the crystal structure of complex **1**
Thermal ellipsoids drawn at the 50% probability level, All H atoms and solvent water molecules are omitted for clarity, Symmetry code: ⁱ $-x, -y, -z$

Fig.1 View of the crystal structure of complex **1**

There exist lots of hydrogen-bond interactions in crystal structure of complex **1** (Fig.2, Table 3). The intermolecular hydrogen-bond interactions $\text{O}(7)\text{--H}(7\text{A}) \cdots \text{O}(4)^{\text{iv}}$, $\text{O}(6)\text{--H}(6\text{B}) \cdots \text{O}(6)^{\text{iii}}$ and $\text{O}(5)\text{--H}(5\text{A}) \cdots \text{O}(2)^{\text{ii}}$ make the dinuclear $\text{Ni}(\text{II})$ units extend along three different directions, i.e. $[110]$, the a and c axes, respectively, leading to a three-dimensional (3D) supramolecular architecture. Furthermore, the lattice aqua molecules play dual roles as both donor and acceptor to construct hydrogen-bonding interactions

with the dinuclear $\text{Ni}(\text{II})$ units. Each lattice aqua molecule is connected with three dinuclear $\text{Ni}(\text{II})$ units through the $\text{O}(6)\text{--H}(6\text{A}) \cdots \text{O}(8)^{\text{iii}}$, $\text{O}(7)\text{--H}(7\text{B}) \cdots \text{O}(8)^{\text{iv}}$, $\text{O}(8)\text{--H}(8\text{A}) \cdots \text{O}(5)^{\text{v}}$ and $\text{O}(8)\text{--H}(8\text{B}) \cdots \text{O}(7)$ hydrogen-bonds for O(8), the $\text{O}(5)\text{--H}(5\text{B}) \cdots \text{O}(9)^{\text{i}}$, $\text{O}(6)\text{--H}(6\text{B}) \cdots \text{O}(9)^{\text{iii}}$, $\text{O}(9)\text{--H}(9\text{B}) \cdots \text{O}(4)^{\text{vi}}$ and $\text{O}(9)\text{--H}(9\text{B}) \cdots \text{O}(3)^{\text{vi}}$ hydrogen-bonds for O(9), respectively, which ulteriorly stabilize the 3D supramolecular structure of **1**.



All H atoms are omitted for clarity. Symmetry codes: ⁱ $-x, -y, -z$; ⁱⁱ $x, y, -1+z$; ⁱⁱⁱ $1-x, -y, -z$; ^{iv} $1-x, 1-y, -z$; ^v $1+x, y, z$; ^{vi} $1-x, 1-y, 1-z$

Fig.2 3D Supramolecular network formed by hydrogen-bonding interactions in **1**

2.2 Magnetic property

The temperature-dependent magnetic susceptibility data of complex **1** have been measured for polycrystalline samples in the temperature range of 1.8~300 K under a 2 000 Oe applied magnetic field, as shown in Fig.3.

At 300 K, the $\chi_{\text{M}}T$ value of **1** is $2.36 \text{ cm}^3 \cdot \text{K} \cdot \text{mol}^{-1}$,

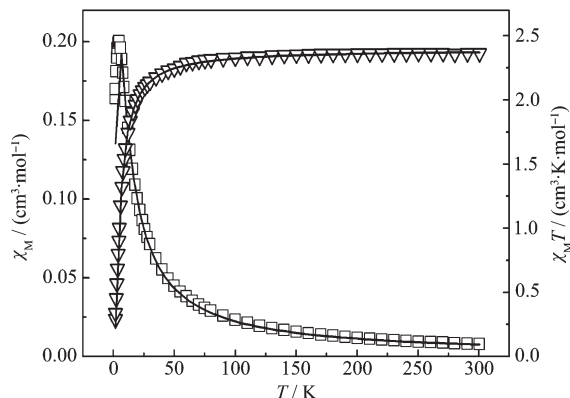


Fig.3 Temperature dependences of the χ_{M} (\square) and $\chi_{\text{M}}T$ (∇) curves for complex **1**

which is higher than the expected value of $2\text{ cm}^3\cdot\text{K}\cdot\text{mol}^{-1}$ for two isolated nickel(II) $S=1$ species with a g factor of 2. This difference might be caused by the spin-orbit coupling characteristic for nickel(II) complexes with an $^3A_{2g}$ ground state resulting in an increasing g factor^[24-25]. While the temperature decreases, the $\chi_{\text{M}}T$ values remain roughly constant down to 60 K and then it decreases rapidly and reaches minimum of $0.29\text{ cm}^3\cdot\text{K}\cdot\text{mol}^{-1}$ at 1.8 K, indicating dominant antiferromagnetic interactions in the complex. Since the shortest Ni \cdots Ni distance between the dinuclear Ni(II) units is 0.6241(1) nm and is long for any significant magnetic interactions, the magnetic Ni(II) \cdots Ni(II) exchange interaction should primarily exist within the dinuclear Ni(II) unit coupled through the triazole nitrogen atoms. The experimental data for **1** were modeled in the range from 300 K to 1.8 K using Equation (1) deduced from the Hamiltonian $H=-2JS_1S_2$ for a simple dinuclear cluster model, where J is the exchange interaction between nickel(II) ions in the dinuclear units, S_i the spin operator for each $S=1$ Ni(II)^[26]. A satisfactory fit was obtained with the parameters $g=2.15$, $J=-1.53\text{ cm}^{-1}$ (with an agreement factor $R=[(\chi_{\text{M}}T)_{\text{calcd}}-(\chi_{\text{M}}T)_{\text{obsd}}]^2/(\chi_{\text{M}}T)_{\text{obsd}}^2=1\times 10^{-5}$), confirming the antiferromagnetic interactions. The g values are reasonable, falling near the range 2.0~2.4 for octahedral nickel(II)^[27]. Antiferromagnetic exchange ranging from weak to moderate is quite frequently observed in dinuclear Ni(II) and Cu(II) systems and trinuclear Cu(II) systems^[28-30].

$$\chi = \frac{2N_{\text{A}}g^2\beta^2}{kT} \times \frac{e^{2J/(kT)} + 5e^{6J/(kT)}}{1 + 3e^{2J/(kT)} + 5e^{6J/(kT)}} \quad (1)$$

2.3 Thermal property

TGA measurements were performed for crystalline samples of **1** in the temperature range of 25~750 °C (Fig.4). A weight loss of 30.43% in 25~250 °C corresponds to the release of six coordinated water molecules and four lattice water molecules and is in agreement with calculated value (29.64%) for complex **1**. From 250~450 °C, the further weight loss is ascribed to the combustion of the organic ligands Htda²⁻. The final product is NiO. At 750 °C, the residual weight of 23.17% is slight lower than the calculated value (24.58%).

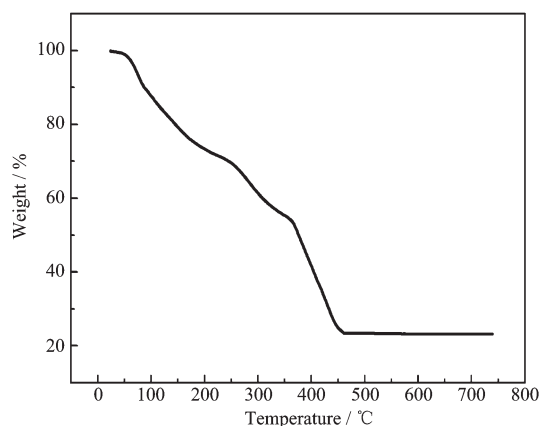


Fig.4 Thermogravimetric curve for **1**

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