以 3-硝基邻苯二甲酸根构建的环状双核 Zn(II), Cd(II)配合物的合成及晶体结构

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摘要:以 3-硝基邻苯二甲酸和咪唑及 2,2′-联吡啶为配体构筑了 2 种配合物[$Z_{n_2(npa)_2(Im)_4}$] (1)和[$Z_{n_2(npa)_2(2,2'-bipy)_2(H_2O)_2$]·2 $Z_{n_2(npa)_2(1m)_4}$] (1)和[$Z_{n_2(npa)_2(2,2'-bipy)_2(H_2O)_2$]·2 $Z_{n_2(npa)_2(1m)_4}$] (1)和[$Z_{n_2(npa)_2(1m)_4}$] (2)加[$Z_{n_2(npa)_2(1m)_4}$] (3)加[$Z_{n_2(npa)_2(1m)_4}$] (4)加[$Z_{n_2(npa)_2(1m)_4}$] (4)加[$Z_{n_2(npa)_2(1m)_4}$] (4)加[$Z_{n_2(npa)_2(1m)_4}$] (4)加[$Z_{n_2(npa)_2(1m)_4}$] (5)加[$Z_{n_2(npa)_2(1m)_4}$] (6)加[$Z_{n_2(npa)_2(1m)_4}$] (6)加[$Z_{n_2(npa)_2(1m)_4}$] (6)加[$Z_{n_2(npa)_2(1m)_4}$] (6)加[$Z_{n_2(npa)_2(1m)_4}$] (7)加[$Z_{n_2(npa)_2(1m)_4}$] (7)加[$Z_{n_2(npa)_4}$] (7)加[$Z_{n_2(npa)_4}$

关键词: 3-硝基邻苯二甲酸; 咪唑; 2,2'-联吡啶; 锌; 镉; 晶体结构

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Synthesis, Crystal Structures of Cyclic Dinuclear Zn(II) and Cd(II) Complexes with 3-Nitrophthalic Acid

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Abstract: Two complexes $[Zn_2(npa)_2(Im)_4]$ (1) and $[Cd_2(npa)_2(2,2'-bipy)_2(H_2O)_2] \cdot 2H_2O$ (2) $(H_2npa=3-nitrophthalic acid, Im=imidazole, 2,2'-bipy=2,2'-bipyridine) were constructed by the use of 3-nitrophthalic acid and imidazole or 2,2'-bipyridine, respectively. The complexes were characterized by elemental analysis, IR, and the crystal structures were determined by single X-ray diffraction. The thermal stabilities of the complexes were studied by thermal gravimetric analysis. The two complexes are dinuclear structures, and both contain <math>M_2C_8O_4$ 14-membered rings. The dinuclear units extend to 3D network through intermolecular hydrogen bonding interactions for complex 1, while a 2D sheet structure is formed for complex 2 by hydrogen bonding and π - π stacking interactions. CCDC: 965024, 1; 965025, 2.

Key words: 3-nitrophthalic acid; imidazole; 2,2'-bipyridine; Zn²⁺; Cd²⁺; crystal structure

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0 Introduction

Supramolecular complexes have received considerable attention not only due to their significance in modern coordination chemistry and crystal engineering but also due to their potential applications such as catalysis, separation, luminescence, and magnetism^[14]. Proper selection of organic ligands and metal ions is a key issue in designing and self-assembly of new functional supramolecular complexes. o-Phthalic acid is a good candidate for the construction of new supramolecular complexes because i) it has diverse coordination modes; and ii) it has different hydrogenbonding modes which depends on the completely or partially deprotonated. Meanwhile, imidazole and 2,2'bipy are widely used as N-donate ligands not only because of their strong coordination abilities but also because of their hydrogen bonding and π - π stacking which are important interactions in construction of supramolecules^[5-10]. However, as we know, only six transition metal complexes with 3-nitrophthalic acid are reported: $[K_2Cu(npa)_2(H_2O)_4]^{[11]}$, $[Cu(npa)(phen)_2]$. H₂npa · H₂O [12], $[Cu (Hnpa)_2 (4,4' -bipy)]_n \cdot 2nH_2O^{[13]},$ $[Zn_2(npa)_2(2,2'-bipy)_2(H_2O)_2] \cdot 2H_2O^{[14]}$, $[Cu_2(npa)_2(2,2'-bipy)_2(2,$ bipy)₂(H₂O)₂] \cdot 2H₂O^[12,15], and [Cu₂(npa)₂(phen)₂(H₂O)₂] \cdot 2H₂O^[16]. Therefore, we are interested in M-npa-Im and M-npa-bipy systems to explore new functional supramolecular complexes. Herein, we report the synthesis and crystal structures of two cyclic dinuclear complexes $[Zn_2(npa)_2(Im)_4]$ (1) and $[Cd_2(npa)_2(2,2'-bipy)_2(H_2O)_2]$. 2H₂O (2). The two complexes are extended to 3D and 2D network by hydrogen bonding and π - π stacking interactions, respectively.

1 Experimental

1.1 Materials and measurements

All reagents and solvents were used as purchased without further purification. IR spectra were recorded on a Perkin-Elmer spectrum one spectrometer with KBr pellet in the region of 4 000~450 cm⁻¹. Elemental analyses of C, H and N were performed on Vario EL III apparatus. Thermogravimetric analysis (TG) data were collected on a Perkin-Elmer Diamond TG/DTA

instrument under nitrogen atmosphere with a heating rate of 10 $^{\circ}\text{C}\cdot\text{min}^{-1}$.

1.2 Synthesis of the complexes

Synthesis of complex [Zn₂(npa)₂(Im)₄] (1): An agueous solution (6 mL) of 3-nitrophthalic acid (50 mg, 0.24 mmol) and NaHCO₃ (40 mg, 0.48 mmol) was added into a H₂O/EtOH solution (8 mL, V_{EiOH} : $V_{\text{H.O}}$ =1: 1) of $Zn(NO_3)_2 \cdot 6H_2O$ (90 mg, 0.30 mmol) with stirring at room temperature. To the resulting solution was added an ethanolic solution (6 mL) of imidazole (35 mg, 0.51 mmol). This mixture was stirred for 30 min at room temperature, then sealed into a 25 mL Teflonlined stainless autoclave under autogenous pressure at 120 °C for 3 d. After cooling to room temperature, light yellow stick crystals were collected by filtration and washed with water (Yield 65 mg, 66%). Anal. Calcd. for 1 C₁₄H₁₁N₅O₆Zn (%): C, 40.94; H, 2.70; N, 17.05. Found (%): C, 41.10; H, 2.67; N, 17.46. IR (KBr, cm⁻¹): 3 267(m), 3 155(m), 3 057(w), 2 954(w), 2 864(w), 1 635(vs), 1 618(w), 1 606(sh), 1 562(w), 1537(s), 1500(w), 1456(s), 1441(sh), 1419(w), 1387 (vs), 1 365(s), 1 346(vs), 1 329(sh), 1 269(m), 1 261 (m), 1 201(w), 1 188(w), 1 173(w), 1 157(w), 1 141(w), 1 128(w), 1 101(m), 1 076(s), 1 066(sh), 955(m), 922(m), 872(w), 849(m), 831(m), 783(m), 769(m), 760(m), 752 (m), 715(s), 656(s), 625(m), 594(w), 573(w), 557(w).

Synthesis of complex [Cd₂(npa)₂(2,2'-bipy)₂(H₂O)₂] · 2H₂O (**2**): The synthetic procedure of **2** is identical to that of **1**, with Cd(NO₃)₂ · 6H₂O (80 mg, 0.26 mmol) instead of Zn(NO₃)₂ · 6H₂O, and 2,2'-bipy (40 mg, 0.26 mmol) instead of imidazole. Colorless block crystals were obtained (Yield 70 mg, 57%). Anal. Calcd. for **2** C₁₈H₁₅N₃O₈Cd (%): C, 42.08; H, 2.94; N, 8.18. Found (%): C, 42.21; H, 2.86; N, 8.29. IR (KBr, cm⁻¹): 3 454 (w), 1 587 (w, vs), 1 549 (sh), 1 520 (vs), 1 491 (m), 1 475 (s), 1 458 (s), 1 439 (s), 1 394 (w, vs), 1 348 (w, vs), 1 319 (sh), 1 296 (m), 1 250 (m), 1 217 (w), 1 180 (m), 1 171 (m), 1 157 (m), 1 130 (w), 1 115 (w), 1 099 (w), 1 072 (w), 1 061 (w), 1 041 (w), 1 016 (s), 974 (w), 926 (s), 831 (s), 768 (vs), 756 (sh), 737 (s), 717 (s), 650 (m), 627 (m), 584 (w), 553 (w).

1.3 Crystal structure determination

Crystallographic data of the two complexes were

collected at room temperature on a Bruker Smart Apex II CCD diffractometer equipped with a graphite -monochromated Mo $K\alpha$ radiation (λ =0.071 073 nm) by using an φ - ω scan mode at 296(2) K. The crystal structures were solved by Patterson and direct methods for complex 1 and 2, respectively, using the program SHELXS-97^[17], and refined on F^2 by full-matrix least-squares techniques using the SHELXTL-97^[18] crystall-ographic software package. All non-hydrogen atoms

were refined anisotropically. The hydrogen atoms of complex 1 were positioned geometrically and refined as riding mode. The hydrogen atoms of complex 2 were tentatively located in difference Fourier maps and refined isotropically. Crystallographic data for both complexes are listed in Table 1, and selected bond lengths and angles are listed in Table 2.

CCDC: 965024, 1; 965025, 2.

Table 1 Crystallographic data for the complexes 1 and 2

Complex	1	2
Empirical formula	$C_{14}H_{11}N_5O_6Zn$	$\mathrm{C_{18}H_{15}N_{3}O_{8}Cd}$
Formula weight	410.65	513.73
Temperature / K	296(2) K	296(2) K
Crystal system	Triclinic	Triclinic
Space group	$P\overline{1}$	$P\bar{1}$
a / nm	0.806 4(2)	0.762 1(4)
b / nm	0.951 7(2)	1.057 3(5)
c / nm	1.118 6(2)	1.247 3(6)
α / (°)	93.475(2)	88.810(5)
β / (°)	105.891(2)	75.379(5)
γ / (°)	99.068(2)	76.658(5)
V / nm^3	0.810 5(3)	0.945 5(8)
Z	2	2
$D_{\rm c}$ / $({ m g} \cdot { m cm}^{-3})$	1.683	1.805
μ / mm ⁻¹	1.560	1.209
F(000)	416	512
Crystal size	0.15×0.12×0.08	0.20×0.15×0.10
θ / (°)	2.67~25.99	2.55~27.48
Reflections collected	6 421	8 327
Independent reflections $(R_{\rm int})$	3 148 (0.014 2)	4 241 (0.018 8)
Parameters	244	294
GOF on F^2	1.102	1.006
R_1 , wR_2 ($I > 2\sigma(I)$)	0.026 2, 0.071 4	0.017 5, 0.047 1
R_1 , wR_2 (all data)	0.028 2, 0.072 6	0.018 4, 0.047 8

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

		1			
Zn(1)-O(1)	0.197 8(2)	Zn(1)-O(3)i	0.197 1(1)	Zn(1)-N(1)	0.199 8(1)
Zn(1)-N(3)	0.201 8(1)				
$O(1)$ - $Zn(1)$ - $O(3)^{i}$	105.14(6)	O(1)-Zn(1)-N(1)	118.80(6)	O(1)-Zn(1)-N(3)	97.98(6)
$N(1)\text{-}Zn(1)\text{-}O(3)^i$	110.39(6)	N(1)- $Zn(1)$ - $N(3)$	103.16(7)	$O(3)^{i}$ - $Zn(1)$ - $N(3)$	121.77(6)
		2			
Cd(1)-O(1)	0.226 6(1)	Cd(1)-O(4)i	0.219 5(1)	Cd(1)-O(7)	0.230 0(1)
Cd(1)-N(1)	0.232 4(2)	Cd(1)- $N(2)$	0.233 2(2)		

Continued Table	2				
O(1)-Cd(1)-O(2)	53.60(5)	O(1)-Cd(1)-O(4)i	101.83(6)	O(1)-Cd(1)-O(7)	97.08(6)
O(1)- $Cd(1)$ - $N(1)$	99.53(6)	$\mathrm{O}(1)\text{-}\mathrm{Cd}(1)\text{-}\mathrm{N}(2)$	163.04(5)	$\mathrm{O}(2)\text{-}\mathrm{Cd}(1)\text{-}\mathrm{O}(4)^{\mathrm{i}}$	149.47(5)
O(2)- $Cd(1)$ - $O(7)$	82.19(6)	O(2)- $Cd(1)$ - $N(1)$	85.16(5)	$\mathrm{O}(2)\text{-}\mathrm{Cd}(1)\text{-}\mathrm{N}(2)$	110.63(5)
N(1)-Cd(1)-N(2)	70.69(6)	$N(1)\text{-}Cd(1)\text{-}O(4)^i$	119.85(5)	N(1)-Cd(1)-O(7)	147.13(5)
N(2)-Cd(1)-O(4) ⁱ	95.10(6)	N(2)-Cd(1)-O(7)	85.65(6)	$O(4)^{i}$ - $Cd(1)$ - $O(7)$	83.74(6)

Symmetry codes: 1: -x+2, -y+2, -z+2; 2: -x+1, -y+1, -z.

2 Results and discussion

2.1 IR spectra

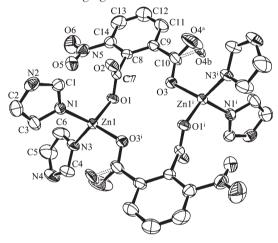
The strong vibrations at 1 606, 1 387 and 1 537, 1 346 cm⁻¹ in complex **1**, and 1 593, 1 390 and 1 549, 1 348 cm⁻¹ in complex **2**, are attributed to the asymmetric and symmetric stretching vibrations of carboxyl and nitryl groups, respectively, indicating that 3nitrophthalic acid exists in complex 1 and 2. The characteristic of C-H bending vibrations are observed in the region of $769 \sim 752$ cm⁻¹ in complex 1, and 768~717 cm⁻¹ in complex 2. The weak vibrations in 3 267~2 864 cm⁻¹ region in complex 1 are assigned to the N-H vibration and hydrogen bonding interactions, and the stretching vibrations of C=C and C=N are observed at 1 635 and 1 618 cm⁻¹, respectively, indicating that imidazole molecule exists in complex 1^[13]. The wide band at 3 454 cm⁻¹ in complex 2 is assigned to stretching vibrations of O-H, indicating that the existence of water molecules. The characteristic bands ν (C=C) and ν (C=N) of 2,2'-bipy are observed at 1 520 and 1 458 cm⁻¹ in complex 2, and the δ (C-H) band is overlapped in 768~717 cm⁻¹ region.

2.2 Crystal structures

2.2.1 Crystal structure of complex [Zn₂(npa)₂(Im)₄] (1)

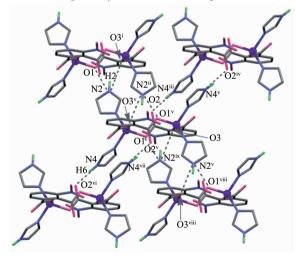
The asymmetric unit of complex ${\bf 1}$ is comprised of the half of a centrosymmetric $[Zn_2(npa)_2(Im)_4]$ structure. As shown in Fig.1, in the dinuclear $[Zn_2(npa)_2(Im)_4]$ structure, there are crystallographically independent one Zn^{2+} ion, one npa^{2-} ligand, and two imidazole molecules. The Zn^{2+} ion is coordinated in a distorted tetragonal geometry with two oxygen atoms from two npa^{2-} ligands (Zn1-O1 0.197 8(2) nm, Zn1-O3 i 0.197 1 (1) nm), and two nitrogen atoms from two imidazole molecules (Zn1-N1 0.199 8 (1) nm, Zn1-N3 0.201 8 (1) nm). Two Zn^{2+} ions are bridged by two

 $\rm npa^{2-}$ ligands with the Zn-Zn distance of 0.536 3 nm, with the form-ation of a $\rm Zn_2C_8O_4$ 14-membered ring. Two carboxyl groups of each $\rm npa^{2-}$ ligand adopt monodentate bridging coordination mode.



Hydrogen atoms are omitted for clarity; symmetry code: i -x+2, -y+2, -z+2

Fig.1 Crystal structure of complex 1



Hydrogen atoms except that involved in hydrogen-bonding interactions are omitted for clarity; symmetry codes: i -1+x, y, z; ii 1-x, 2-y, 2-z; iii x, 1+y, z; iv 2-x, 3-y, 2-z; v 2-x, 2-y, 2-z; vi x, -1+y, z; vi 2-x, 1-y, 2-z; vii 3-x, 2-y, 2-z; ix 1+x, y, z

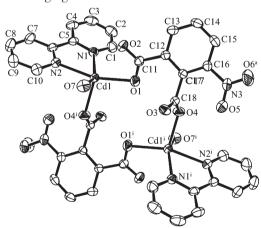
 $\label{eq:Fig.2} Fig. 2 \quad \text{Intermolecular hydrogen-bonding interactions} \\ \quad \text{around each } [Zn_2(npa)_2(Im)_4] \text{ unit in complex } \mathbf{1}$

In the crystal lattice, each $[Zn_2(npa)_2(Im)_4]$ unit is hydrogen bonded with four adjacent ones, two through two pairs of N4–H···O2 hydrogen bonds (N4–H6···O2 0.282 3(2) nm), and the other two through two pairs of N2–H···O1 and N2–H···O3 hydrogen bonds (N2–H2···O1 0.292 1 (2) nm, N2–H2···O3 0.299 4 (2) nm), creating a 3D hydrogen bonding network structure.

2.2.2 Crystal structure of complex

$[Cd_2(npa)_2(2,2'-bipy)_2(H_2O)_2] \cdot 2H_2O$ (2)

The single-crystal X-ray diffraction analysis revealed that complex 2 is a dinuclear structure. It contains one Cd²⁺ ion, one npa²⁻ ligand, one 2,2'-bipy molecule, one coordination and one crystallization water molecules in the asymmetric unit. As shown in Fig.3, the Cd²⁺ ion is five coordination in a distorted trigonal bipyramidal geometry with two oxygen atoms from two npa²⁻ ligands (Cd1-O1 0.226 6(1) nm, Cd1-O4i 0.219 5(1) nm), two nitrogen atoms from one bipyridine molecule (Cd1-N1 0.232 4(2) nm, Cd1-N2 0.233 2 (2) nm), and one water molecule (Cd1-O7 0.230 0 (1) nm). Two Cd2+ ions are bridged by two npa²⁻ ligands with the Cd-Cd distance of 0.569 4 nm, with the formation of a Cd₂C₈O₄ 14-membered ring. Two carboxyl groups of each npa²⁻ ligand adopt monodentate bridging coordination mode.



Hydrogen atoms and crystallization water molecules are omitted for clarity; symmetry code: i -x+1, -y+1, -z

Fig.3 Crystal structure of complex 2

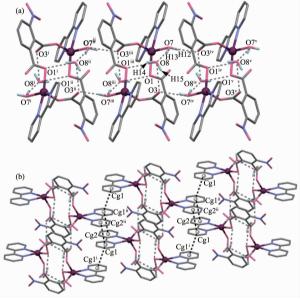
The crystallization water molecule is intramolecular hydrogen-bonded with a coordination oxygen, an uncoordination oxygen of two carboxyl groups, and coordination water molecule (O8–H14····

O1 0.289 7(3) nm, O8-H15···O3 0.294 7(2) nm, O7-H13···O8 0.273 3 (3) nm). In addition, the coordination water molecule is intermolecular hydrogen-bonded with an uncoordination oxygen atom of neighboring molecule (O7-H12···O3 0.280 1(2) nm). Thus, a 1D chain structure is created. The neighboring chains are extended to a 2D sheet structure with π - π stacking interactions between bipyridine molecules (centroid-to-centroid distances of 0.350 6(2) and 0.360 3(2) nm, respectively, see Table 3 and Fig.4).

Table 3 π - π interactions in complex 2

Ring	Distance / nm	Dihedral angle / (°)
Cg1→Cg1 ⁱ	0.350 6(2)	0
Cg1→Cg2 ⁱⁱ	0.360 3(2)	3.76(10)
Cg2→Cg1 ⁱⁱ	0.360 3(2)	3.76(10)

Ring: Cg1: 6-Membered Ring N1-C1-C2-C3-C4-C5; Cg2: 6-Membered Ring N2-C6-C7-C8-C9-C10; Symmetry codes: i 1 -x, 1-y, 1-z; ii -x, 1-y, 1-z.



Hydrogen atoms except that involved in hydrogen-bonding interactions are omitted for clarity; symmetry codes: i 2-x, 1-y, -z; ii 1+x, y, z; iii 1-x, 1-y, -z; iv -x, 1-y, -z; v -1+x, y, z

Fig.4 (a) 1D hydrogen-bonding chain structure; (b) 2D sheet structure constructed by hydrogen-bonding and π - π stacking interactions of complex 2

2.3 TGA

The thermogravimetric analyses were carried out to examine the thermal stability of the two complexes (Fig.5). For complex 1, the first weight loss of 41.80% between 190 and 240 $^{\circ}$ C is attributed to the loss of

1.6 npa²⁻ ligands (Calcd. 40.74%). Both the second and third degradation stages correspond to the loss of two imidazole molecules (Calcd. 16.58%), which are in the range of 240~292 °C and 292~450 °C, with the mass loss of 16.13% and 14.82%, respectively. For complex **2**, the first weight loss of 7.29% in the range of 90~120 °C corresponds to the loss of four H₂O molecules (Calcd. 7.01%). The second stage occurs in the range of 195~420 °C with the loss of 48.91%, corresponding to the loss of two 3-npa²⁻ ligands and a half of 2,2′-bipy molecule (Calcd. 48.30%). The remaining component [Cd₂(2,2′-bipy)_{1.5}]⁴⁺ starts to decompose above 640 °C, and it does not end at the temperature limit.

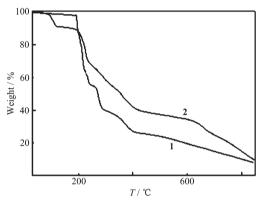


Fig.5 TG curves of complexes 1 and 2

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