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## Hydrothermal synthesis and crystal structure of Cd(II) complex: [[Cd<sub>2</sub>(L<sup>1</sup>)<sub>2</sub>Cl<sub>4</sub>(H<sub>2</sub>O)<sub>2</sub>](L<sup>2</sup>)<sub>2</sub>][HL<sup>1</sup>=3-(2-pyridyl)pyrazole, L<sup>2</sup>=5-nitro-1,3-benzenedicarboxylic acid]

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### ABSTRACT

One new Cd(II) dinuclear complex: {[Cd<sub>2</sub>(L<sup>1</sup>)<sub>2</sub>Cl<sub>4</sub>(H<sub>2</sub>O)<sub>2</sub>](L<sup>2</sup>)<sub>2</sub>}[HL<sup>1</sup>=3-(2-pyridyl)pyrazole, L<sup>2</sup>=3-Nitro-1,2-benzenedicarboxylic acid] was prepared by hydrothermal reaction and structurally characterized. Single crystal X-ray diffraction analysis showed that the Cd(II) lies in distorted octahedron coordination geometry. Crystal data: triclinic, space group P-1, a = 7.4811(15) Å, b = 10.741(2) Å, c = 11.480(2) Å, α = 87.44(3)°, β = 86.71(3)°, γ = 85.69(3)°, V = 917.6(3) Å<sup>3</sup>, Z = 1, D = 2.070 Mg·m<sup>-3</sup>. © 2008 Trade Science Inc. - INDIA

### KEYWORDS

Hydrothermal synthesis;  
Single crystal;  
Cd(II) complex.

### INTRODUCTION

In the recent years, the synthesis of multi-nuclear and discrete or infinite coordination polymers has attracted great interest in coordination chemistry due to their intriguing topologies and potential applications as functional materials<sup>[1-2]</sup>. Rational design and synthesis coordination architectures (discrete or infinite) with organic ligands and metal ions is still a long term challenge. Carefully select organic ligand and metal ions are the main keys of crystal engineering. 3-(2-Pyridyl)pyrazole (HL<sup>1</sup>) as a multifunctional ligand with different coordination modes, was first reported by Tisler and co-workers<sup>[3]</sup>. After that, HL<sup>1</sup> was just considered as a 2,2'-bipyridine like bidentate ligand until 1996, Ward and coworkers observed another coordination mode, acting as a terdentate bridging ligand via deprotonation of the NH group and which can coordinate to a second metal ion<sup>[4]</sup>. A triple-stranded helical complex has been reported by Lam and co-workers<sup>[5]</sup>. Therefore, the coordination chemistry of HL<sup>1</sup> with some transition metal ions, has been studied by several researchers<sup>[6-7]</sup>. However, to the best of our knowledge, the Cd(II) complexes with HL<sup>1</sup> are reported rarely. Herein, we report the hydrothermal synthesis of a new Cd(II) com-

plex with ligand HL<sup>1</sup>.

### EXPERIMENTAL

#### 1. Synthesis of {[Cd<sub>2</sub>(L<sup>1</sup>)<sub>2</sub>Cl<sub>4</sub>(H<sub>2</sub>O)<sub>2</sub>](L<sup>2</sup>)<sub>2</sub>}

A mixture of CdCl<sub>2</sub> (0.2mmol), 3-(2-pyridyl)pyrazole (0.2mmol), 5-nitro-1,3-benzenedicarboxylic acid (0.1mmol), and distilled water (10 mL) was sealed in a 25mL stainless steel reactor with Teflon liner and directly heated to 120°C for two days, then cooled to room temperature during 24 h. Block single crystals were obtained in 20% yield.

#### 2. Structure determination

X-ray single-crystal diffraction data for the cobalt complex was collected on a Bruker Smart 1000 CCD diffractometer at 293(2) K with Mo-Kα radiation (= 0.71073 Å) by scan mode. The program SAINT<sup>[8]</sup> was used for integration of the diffraction profiles. Semiempirical absorption corrections were applied using SADABS program. All the structures were solved by direct methods using the SHELXS program of the SHELXTL package and refined by full-matrix least-squares methods with SHELXL<sup>[9]</sup> Metal atoms in each complex were located from the E-maps and other non-

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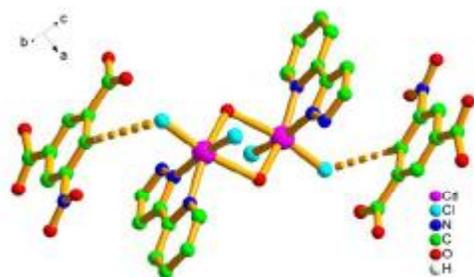


Figure 1: View of the structure of Cd(II) in the complex

TABLE 1: Crystallographic data and structure refinement summary for the complex

Formula	C <sub>32</sub> H <sub>20</sub> Cd <sub>2</sub> Cl <sub>4</sub> N <sub>8</sub> O <sub>14</sub>		
Formula weight	1105.14	$\gamma$ / deg	85.69(3)
Crystal system	Triclinic	V / Å <sup>3</sup>	917.6(3)
Space group	P-1	Z	1
a (Å)	7.4811(15)	D / g cm <sup>-3</sup>	2.000
b (Å)	10.741(2)	$\mu$ / mm <sup>-1</sup>	1.533
c (Å)	11.480(2)	T / K	293(2)
$\alpha$ / deg	87.44(3)	R(I>2 $\sigma$ )/wR (all data)	0.0773/ 0.2272
$\beta$ / deg	86.71(3)	Total /unique	7796/3146/0.
		/ R <sub>int</sub>	0198

TABLE 2: Selected bond distances (Å) and Angles (deg) for the complex

Cd(1)-Cl(1)	2.070(5)	Cd(1)-Cl(2)	2.070(5)
Cd(1)-O(1W)	2.136(4)	Cd(1)-N(1)	2.141(5)
Cd(1)-N(2)	2.122(5)	Cd(1)-O(1W)#1	2.380(5)
Cl(2)-Cd(1)-Cl(1)	90.29(19)	Cl(2)-Cd(1)-N(2)	93.7(2)
Cl(1)-Cd(1)-N(2)	171.17(19)	Cl(2)-Cd(1)-O(1W)	94.96(19)
Cl(1)-Cd(1)-O(1W)	94.96(19)	N(2)-Cd(1)-O(1W)	95.41(19)

hydrogen atoms were located in successive difference Fourier syntheses and refined with anisotropic thermal parameters on F<sup>2</sup>. Hydrogen atoms of carbon were included in calculated positions and refined with fixed thermal parameters riding on their parent atoms. Crystallographic data and experimental details for structural analysis are summarized in TABLE 1. Selected bond lengths and angles are listed in TABLE 2.

## RESULTS AND DISCUSSION

The title compound was obtained as an air-stable colorless solid after heated to 120°C for two days. Single-crystal analysis reveals that though two different ligands have been used in the experiment, only HL<sup>1</sup> coordinate to the Cd(II) center, while L<sup>2</sup> lies in the crystal lattice as guest molecular. As shown in figure 1, in the complex the Cd(II) adopts a distorted octahedron coordinate geometry, which coordinates to two N atoms of HL<sup>1</sup>, two O atoms of water molecules, and two Cl<sup>-</sup> anions. The distance of Cd–N are 2.117(5) and

2.140(5) Å, the distance of Cd–Cl between 2.069(4) and 2.137(4) Å, and the distance of Cd–O between 2.136(4) and 2.380(5) Å, the angles around Cd(II) are between 90.29(19) and 171.17(19)°, all the distances and angles are in the normal range. It is very interesting that the mononuclear complex forms the dinuclear complex bridging by two Cl<sup>-</sup> anions. L<sup>2</sup> does not coordinate to the metal center, but there are C–H...Cl weak interactions between the H atoms of L<sup>2</sup> and two Cl<sup>-</sup> anions [C(16)–Cl(3) = 3.5654 Å, H(16)–Cl(3) = 2.7639 Å, C(16)–H(16)–Cl(3) = 144.852°].

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