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NOTE

Hydrothermal Synthesis and Crystal Structure of 1,4-Di-pyridin-4-yl-piperazine Camphorate Cadmium(II)

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One new cadmium compound using $Cd(NO_3)_2{\cdot}2H_2O$, camphoric acid and bpp (1,4-dipyridin-4-yl-piperazine) with m.f. $C_{36}H_{48}N_4O_8Cd_2$ has been successfully synthesized. The compound shows a one-dimensional framework. The 3D supramolecular structure is formed *via* hydrogen bonding connection.

Keywords: Coordination polymer, Crystal structure, Cadmium(II).

Metal organic frameworks (MOFs) have received much attention in the field of crystal engineering and supramolecular chemistry because of their diverse structures and promising applications in functional materials such as luminescent materials, gas adsorption and magnetism[1-4]. Hydrogen bonds are well suited for the design of polymeric arrangement and crystal engineering because of their important directional interactions and because they can interlink 1-D, or 3-D structures into higher-dimensionality systems[5,6].

All reagent and solvents employed were commercially available and used as received without further purification.

A mixture of $Cd(NO_3)_2{\cdot}2H_2O$ (0.0401 g), camphoric acid (0.1033 g), bpp (1,4-dipyridin-4-yl-piperazine) (0.2000 g) and distilled water (8.0230 g) was heated in a 25 mL stainless steel reactor with a Teflon liner 413 K for 4 days, followed by slow cooling to room temperature. Colourless crystals of the compound are formed (Fig. 1).

Detection method: Diffraction intensity data of the single crystal of the five compounds were collected on a Bruker SMART APEX-II CCD diffractometer equipped with a graphite monochromated MoK_α radiation ($\lambda = 0.71073$ Å) by using a ω-scan mode. All the structures were solved by direct methods and refined by full-matrix least-squares methods on F^2 using the program SHEXL 97[7]. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms were located by geometrically calculations and their positions and thermal parameters were fixed during the structure refinement. The crystallographic data and experimental details of structural analyses for coordination polymers are summarized in Table-1. Selected bond and angle parameters are listed in Table-2.

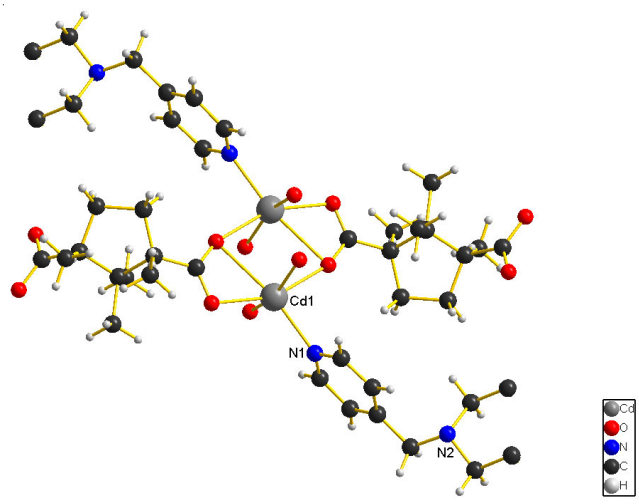

Fig. 1. Molecular structure of the title compound at 30 % probability displacement ellipsoids

X-ray diffraction analysis revealed that the fundamental building unit consists of Cd(II) ion and bpp (1,4-dipyridin-4-yl-piperazine) and camphoric acid as bridging ligands to construct a new coordination polymer. The asymmetric unit of the title structure contains two Cd(II) ion, two camphoric acid and one bpp molecule. The Cadmium atom is six-coordinated in a distorted octahedral manner and one nitrogen atom

TABLE-1
CRYSTALLOGRAPHIC DATA AND STRUCTURE REFINEMENT SUMMARY FOR COMPLEX

Empirical formula	$C_{36}H_{48}N_4O_8Cd_2$
Formula weight	889.58
Crystal system space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 11.87(3) Å; b = 13.75(3) Å; c = 13.20(3) Å
Volume (Å^3)	1959(7)
θ range for data collection	2.25 -25.50
Final R indices [I>2σ (I)]	R_1 = 0.0676, wR_2 = 0.1068
Z, calculated density (mg/m^3)	2, 1.508
Absorption coefficient (mm^{-1})	1.137
F(000)	904
Limiting indices	-14≤h≤14; -16≤k≤16; -15≤l≤15
Largest diff. peak and hole (e/Å^3)	0.784 and -1.098
Goodness of fit on F^2	1.129
R indices (all data)	R_1= 0.1668, wR_2= 0.1302

TABLE-2
SELECTED BOND LENGTHS (Å) AND ANGLES (°) FOR COMPLEX

Cd(1)-N(1)	2.214(8)	Cd(1)-O(1)	2.368(7)
Cd(1)-O(3)#1	2.190(7)	Cd(1)-O(2)#3	2.239(7)
Cd(1)-O(4)#2	2.193(7)	Cd(1)-O(1)#3	2.556(7)
O(3)#1-Cd(1)-O(4)#2	158.9(2)	N(1)-Cd(1)-O(1)	87.2(3)
O(3)#1-Cd(1)-N(1)	95.5(3)	O(2)#3-Cd(1)-O(1)	157.6(2)
O(4)#2-Cd(1)-N(1)	100.0(3)	O(3)#1-Cd(1)-O(1)#3	84.3(2)
O(3)#1-Cd(1)-O(2)#3	94.7(2)	O(4)#2-Cd(1)-O(1)#3	83.3(2)
O(4)#2-Cd(1)-O(2)#3	91.7(3)	N(1)-Cd(1)-O(1)#3	168.9(2)
N(1)-Cd(1)-O(2)#3	115.1(3)	O(2)#3-Cd(1)-O(1)#3	53.9(3)
O(3)#1-Cd(1)-O(1)	82.2(3)	O(1)-Cd(1)-O(1)#3	103.7(2)
O(4)#2-Cd(1)-O(1)	84.2(2)	N(1)-Cd(1)-Cd(1)#3	141.8(2)

Symmetry codes: #1 x,-y+1/2,z+1/2 #2 -x+1,y-1/2,-z+1/2 #3 -x+1,-y,-z+1

N1 from the bpp ligand. The Cd-N bond lengths is 2.214(8) Å and four oxygen atoms (O1, O2, O3 and O4) from two camphoric acid molecules. The Cd-O bond lengths are 2.368(7) Å, 2.239(7) Å, 2.190(7) Å and 2.193(7)Å, respectively. The chains are further assembled by the intermolecular hydrogen bonding interaction leading to the formation of a 3D framework (Fig. 2).

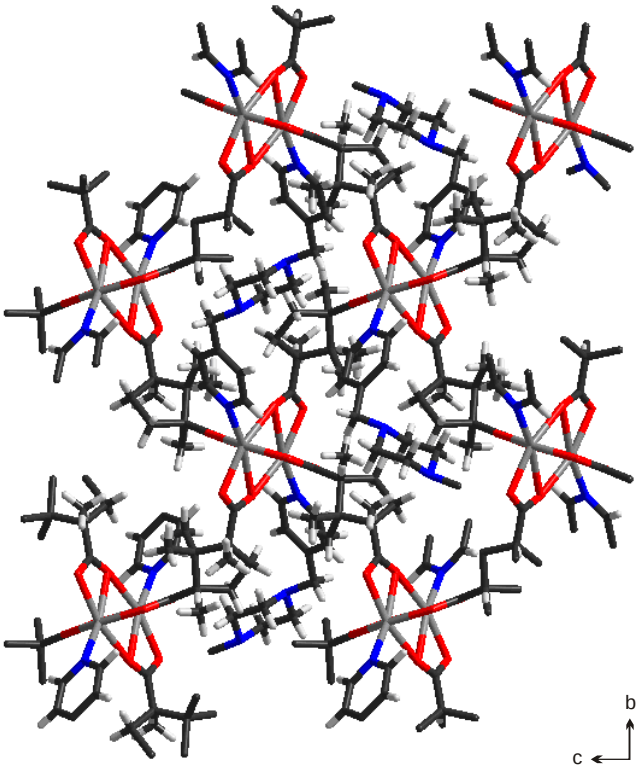

Fig. 2. 3D structure formed *via* hydrogen bonding interactions

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