Lai-Jun Zhang*, Wen-Fang Weng, Shuai Huang, Xiao-Qing Ye, Yuan-Yuan Deng, Jia-Yi Lang, Qing Yin and Jun-Jie Ding

Crystal structure of poly[diaqua-bis(μ$_2$-5-((pyridin-4-yl-methyl)amino)benzene-1,3-dicarboxylato-κ^2N:O)cadmium(II)], $C_{28}H_{26}CdN_4O_{10}$

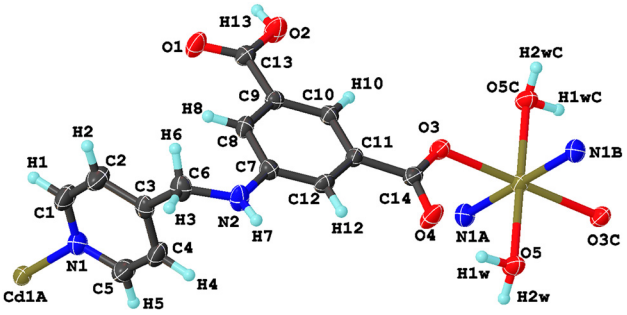

Table 1: Data collection and handling.

Crystal:	Colourless plate
Size:	0.42 × 0.29 × 0.13 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.89 mm^{-1}
Diffractometer, scan mode:	APEX2, omega
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	3529, 2318, 0.017
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2228
$N(param)_{refined}$:	196
Programs:	Bruker,[1] Olex2,[2] SHELX[3,4]

https://doi.org/10.1515/ncrs-2024-0430
Received November 1, 2024; accepted November 29, 2024; published online December 23, 2024

Abstract

$C_{28}H_{26}CdN_4O_{10}$, triclinic, $P\bar{1}$ (no. 2), a = 5.7700(12) Å, b = 10.190(2) Å, c = 12.160(2) Å, α = 70.38(3)°, β = 84.72(3)°, γ = 81.63(3)°, V = 665.6(3) Å^3, Z = 1, $R_{gt}(F)$ = 0.0272, $wR_{ref}(F^2)$ = 0.0602, T = 293 K.

CCDC No.: 1822051

A part of the molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

5-[(Pyridin-4-yl-methyl)amino]isophthalic acid ($C_{14}H_{12}N_2O_4$, H_2L) was synthesized following the literature reported by us.[4]

Both the analytical-grade *N*,*N*-dimethylformamide (DMF) and cadmium chloride hemipentahydrate ($CdCl_2{\cdot}2.5H_2O$) were commercially available and were utilized without further purification. The title compound was solvo/hydrothermally synthesized using the following procedure. 0.2 mmol (0.0544 g) of H_2L, 1 ml of DMF, 5 ml of distilled water and 0.2 mmol (0.457 g) of $CdCl_2{\cdot}2.5H_2O$ were added to a 15 ml Teflon reaction vessel and then sonicated for 10 min, resulting in a white suspension liquid. The Teflon-lined stainless steel reaction vessel was sealed and heated at 110 °C for 136 h. Upon cooling to room temperature, a large amount of colorless rod crystals were obtained with a yield of 73 % based on $CdCl_2{\cdot}2.5H_2O$ after being isolated by filtration, washed with the *N*,*N*-dimethylformamide/water mixture and dried at room temperature.

2 Experimental details

The structure was solved by Direct Methods by SHELXT program and refined by SHELXL program. All H-atoms bound to the C atoms were positioned with idealized geometry and refined isotropically with U_{iso}(H) = 1.2 times U_{eq}(C) using a riding model with C–H = 0.93 Å for aromatic H atoms and C–H = 0.97 Å for methylene H atoms. The H-atoms (H1w, H2w, H13, and H7) from O and N atoms were positioned with Q peaks and refined isotropically with U_{iso}(H) = 1.5 times U_{eq}(O) or U_{iso}(H) = 1.2 times U_{eq}(N).

***Corresponding author: Lai-Jun Zhang**, School of Chemistry and Environmental Science, Shangrao Normal University, Shangrao 334001, China, E-mail: ljzhang@sru.edu.cn. https://orcid.org/0000-0003-2515-712X
Wen-Fang Weng, Editorial Office of Journal of Shangrao Normal University, Shangrao Normal University, Shangrao 334001, China
Shuai Huang, Xiao-Qing Ye, Yuan-Yuan Deng, Jia-Yi Lang, Qing Yin and Jun-Jie Ding, School of Chemistry and Environmental Science, Shangrao Normal University, Shangrao 334001, China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.8514 (5)	0.2559 (3)	1.4358 (3)	0.0419 (8)
H1	0.9513	0.2806	1.4795	0.050*
C2	0.9338 (5)	0.1501 (3)	1.3902 (3)	0.0404 (7)
H2	1.0862	0.1057	1.4031	0.049*
C3	0.7904 (5)	0.1100 (3)	1.3256 (2)	0.0287 (6)
C4	0.5684 (5)	0.1820 (3)	1.3091 (3)	0.0368 (7)
H4	0.4651	0.1594	1.2657	0.044*
C5	0.4991 (5)	0.2868 (3)	1.3565 (3)	0.0382 (7)
H5	0.3485	0.3342	1.3434	0.046*
C6	0.8707 (5)	−0.0073 (3)	1.2751 (2)	0.0326 (6)
H6	1.0406	−0.0191	1.2678	0.039*
C7	0.8460 (4)	0.1228 (3)	1.0647 (2)	0.0244 (6)
C8	1.0193 (4)	0.2042 (3)	1.0678 (2)	0.0263 (6)
H8	1.0959	0.1849	1.1365	0.032*
C9	1.0780 (4)	0.3132 (3)	0.9694 (2)	0.0230 (5)
C10	0.9663 (4)	0.3445 (3)	0.8658 (2)	0.0247 (6)
H10	1.0050	0.4187	0.8005	0.030*
C11	0.7949 (4)	0.2631 (3)	0.8608 (2)	0.0225 (5)
C12	0.7385 (4)	0.1544 (3)	0.9592 (2)	0.0251 (6)
H12	0.6248	0.1005	0.9549	0.030*
C13	1.2613 (4)	0.3961 (3)	0.9810 (2)	0.0268 (6)
C14	0.6641 (4)	0.2959 (3)	0.7513 (2)	0.0261 (6)
Cd1	0.5000	0.5000	0.5000	0.02479 (10)
H1w	0.2765	0.3122	0.6591	0.050*
H2w	0.0670	0.3913	0.6201	0.050*
H3	0.8222	−0.0941	1.3292	0.039*
H7	0.6903	−0.0374	1.1562	0.038*
H13	1.3999	0.5406	0.8981	0.059*
N1	0.6364 (4)	0.3244 (2)	1.42049 (19)	0.0322 (5)
N2	0.7798 (4)	0.0172 (2)	1.16294 (19)	0.0318 (5)
O1	1.3491 (3)	0.3733 (2)	1.07464 (17)	0.0378 (5)
O2	1.3181 (3)	0.4938 (2)	0.88512 (17)	0.0392 (5)
O3	0.7277 (3)	0.39361 (19)	0.66116 (15)	0.0296 (4)
O4	0.5020 (3)	0.2267 (2)	0.75398 (17)	0.0390 (5)
O5	0.1994 (3)	0.36707 (19)	0.58998 (16)	0.0333 (4)

3 Comment

At present, the use of diverse polydentate ligands containing rich N/O coordinating atoms is preferred in the design and synthesis of metal coordination polymers including metal-organic frameworks because they generally can provide multiple coordination sites and exhibit various coordination geometries.[5–7] In the past two decades, as a flexible organic polydentate ligand, 5-[(pyridin-4-yl-methyl)amino]isophthalic acid (H_2L) has been selected to synthesize many interesting metal coordination polymers with some fascinating structural architecture.[8–11] The Cd(II) atom as the coordination center is a usual choice in the construction of metal coordination polymers, however, these reported metal coordination polymers based on H_2L-related ligand, including our previously reported Cd–MOF [Cd(*L*)]·(DMF)(H_2O),[12] are three-dimensional in the crystal structure. Herein, we obtained the first example of one-dimensional Cd(II) coordination polymer when using H_2L as organic ligand through a solvo/hydrothermal method.

The asymmetric unit consists of one half of a Cd(II), one HL^- anion, and one coordinated water molecule. Each Cd(II) atom is six-coordinated with two pyridine nitrogen atoms from two HL^- ligands and four oxygen atoms from another two HL^- ligands and two coordinated water molecules, which results in a slightly distorted octahedral geometric configuration {CdO_4N_2}. The Cd–N and Cd–O lengths are in the ranges of 2.317(2) Å and 2.3182(19)–2.3193(19) Å, respectively. These bond distances are in agreement with these previously reported cadmium coordination polymers.[13] In the title compound, the HL^- ligand serves as a μ_2-bridge linking two adjacent Cd(II) atoms with its pyridine nitrogen atom and one carboxylate oxygen atom, where the dihedral angle between benzene ring and pyridine ring is 116.57°. Pairs of HL^- ligands connect symmetry-related Cd(II) atoms through monodentate carboxylate O and pyridine N atoms with the Cd···Cd separation of 12.160(10) Å to form one-dimensional loop-like chain. An infinite three-dimensional supramolecular structure in the title compound was further formed from the one-dimensional loop-like chains and stabilized through hydrogen bonding and π–π stacking interactions. The distance between the benzene rings of two adjacent HL^- ligands is 3.3599(12)–3.4205(12) Å, indicating the presence of π–π stacking interactions. Rich hydrogen bonding interactions include an intramolecular hydrogen bond (O5–H1w···O4) and intermolecular hydrogen bondings (O5–H2w···O3A, N2–H7···O4B, O2–H13···O1C).

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.
Research funding: College Students Innovation and Entrepreneurship Training Program of Shangrao Normal University (No. 2024-CX-10) and the Science and Technology Research Project of the Education Department of Jiangxi Province (No. 201708).
Conflict of interest: The authors declare no conflicts of interest regarding this article.

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