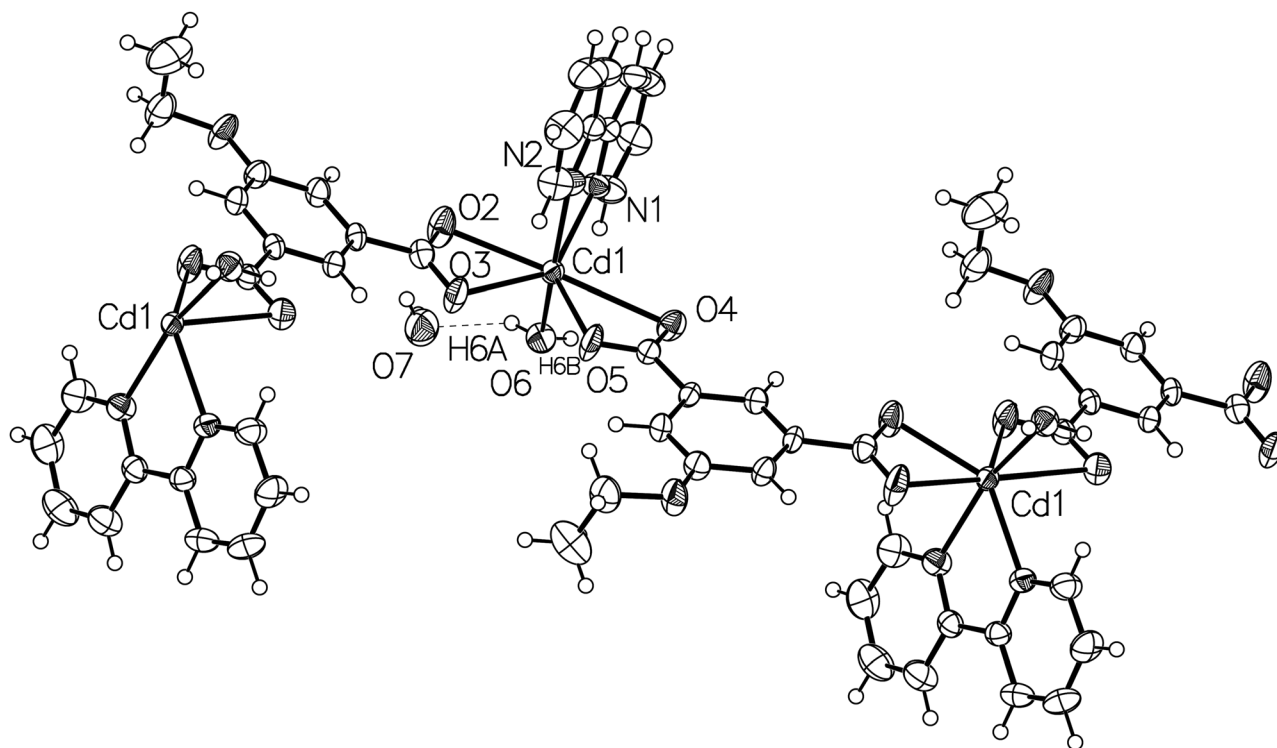


Lilei Zhang*, Deming Chang and Mengdan Dun

The crystal structure of catena-poly[aqua-2,2'-bipyridine- $\kappa^2 N, N'$ -(μ_2 -5-ethoxyisophthalato- $\kappa^4 O, O': O'', O'''$)cadmium(II)] monohydrate, $C_{20}H_{20}CdN_2O_7$



<https://doi.org/10.1515/ncrs-2022-0057>

Received January 28, 2022; accepted April 25, 2022;
published online May 5, 2022

Abstract

$C_{20}H_{20}CdN_2O_7$, monoclinic, $P2_1/c$ (no. 14), $a = 9.0154(3)$ Å, $b = 17.7168(8)$ Å, $c = 12.8449(5)$ Å, $\beta = 98.537(4)^\circ$, $V = 2028.91(14)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0320$, $wR_{ref}(F^2) = 0.0715$, $T = 288.32(10)$ K.

CCDC no.: 2168529

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.20 × 0.18 × 0.15 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.12 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
θ_{max} , completeness:	25.0°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	8050, 3545, 0.026
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 3139
$N(param)_{refined}$:	275
Programs:	CrysAlis ^{PRO} [1], Olex2 [2, 3]

*Corresponding author: Lilei Zhang, College of Chemistry and Chemical Engineering, Luoyang Normal University, Luoyang, China, E-mail: zhanglilei@outlook.com. <https://orcid.org/0000-0003-0798-1991>

Deming Chang and Mengdan Dun, College of Chemistry and Chemical Engineering, Luoyang Normal University, Luoyang, China

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.

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	0.7114 (3)	0.49297 (19)	1.0001 (2)	0.0321 (8)
C2	0.6951 (3)	0.57060 (18)	1.0092 (2)	0.0311 (8)
H2	0.7360	0.5948	1.0710	0.037*
C3	0.6185 (3)	0.61219 (19)	0.9269 (2)	0.0263 (7)
C4	0.5556 (3)	0.57580 (18)	0.8352 (2)	0.0255 (7)
H4	0.5027	0.6035	0.7803	0.031*
C5	0.5710 (3)	0.49862 (17)	0.8247 (2)	0.0248 (7)
C6	0.6511 (3)	0.45757 (19)	0.9075 (2)	0.0312 (7)
H6	0.6640	0.4059	0.9003	0.037*
C7	0.8090 (5)	0.3772 (2)	1.0831 (3)	0.0493 (10)
H7A	0.7213	0.3532	1.0440	0.059*
H7B	0.8242	0.3568	1.1540	0.059*
C8	0.9414 (5)	0.3613 (3)	1.0319 (4)	0.0768 (15)
H8A	0.9272	0.3828	0.9625	0.115*
H8B	0.9544	0.3077	1.0270	0.115*
H8C	1.0288	0.3830	1.0727	0.115*
C9	0.6064 (3)	0.69634 (19)	0.9383 (2)	0.0304 (8)
C10	0.5007 (3)	0.45714 (19)	0.7283 (2)	0.0274 (7)
C11	0.1845 (4)	0.4344 (2)	0.3716 (2)	0.0408 (9)
H11	0.2796	0.4393	0.3525	0.049*
C12	0.0633 (4)	0.4627 (2)	0.3051 (2)	0.0454 (10)
H12	0.0761	0.4861	0.2422	0.054*
C13	−0.0760 (4)	0.4557 (2)	0.3338 (2)	0.0466 (10)
H13	−0.1598	0.4748	0.2908	0.056*
C14	−0.0910 (4)	0.4206 (2)	0.4258 (2)	0.0403 (9)
H14	−0.1856	0.4152	0.4455	0.048*
C15	0.0339 (3)	0.39273 (19)	0.4902 (2)	0.0290 (7)
C16	0.0247 (4)	0.35405 (18)	0.5917 (2)	0.0308 (8)
C17	−0.1104 (4)	0.3420 (2)	0.6289 (3)	0.0478 (10)
H17	−0.2001	0.3582	0.5901	0.057*
C18	−0.1100 (4)	0.3057 (2)	0.7245 (3)	0.0564 (11)
H18	−0.1994	0.2971	0.7506	0.068*
C19	0.0230 (5)	0.2826 (2)	0.7798 (3)	0.0540 (11)
H19	0.0256	0.2578	0.8438	0.065*
C20	0.1530 (4)	0.2965 (2)	0.7395 (2)	0.0460 (10)
H20	0.2435	0.2810	0.7779	0.055*
Cd1	0.37640 (2)	0.35203 (2)	0.57537 (2)	0.02504 (10)
N1	0.1708 (3)	0.40009 (16)	0.46223 (18)	0.0306 (6)
N2	0.1547 (3)	0.33131 (16)	0.64722 (19)	0.0343 (7)
O1	0.7852 (3)	0.45775 (14)	1.08703 (16)	0.0480 (7)
O2	0.6682 (3)	0.72804 (13)	1.01974 (17)	0.0506 (7)
O3	0.5349 (3)	0.73345 (13)	0.86385 (17)	0.0441 (6)
O4	0.4391 (2)	0.49213 (13)	0.64944 (15)	0.0375 (6)
O5	0.5043 (3)	0.38653 (14)	0.73137 (18)	0.0493 (7)
O6	0.5449 (2)	0.38007 (13)	0.46584 (16)	0.0369 (6)
H6A	0.5286	0.3521	0.4100	0.055*
H6B	0.5369	0.4221	0.4314	0.055*
O7	0.5858 (3)	0.26140 (17)	0.3453 (2)	0.0579 (7)
H7C	0.5243	0.2312	0.3674	0.087*
H7D	0.5630	0.2669	0.2791	0.087*

Source of material

The title compound was synthesized by the hydrothermal method. Samples of 5-ethoxisophtalic acid (0.25 mmol) and 2,2'-bipyridine (0.25 mmol) were placed in 8 mL deionized water and stirred for 10 min at ambient temperature. The Cd(NO₃)₂·4H₂O (0.25 mmol) was added to the reaction solution and continuously stirred for 30 min to obtain a uniform solution. The resulting reaction mixture was sealed in a Teflon-lined autoclave and maintained at 120 °C for 48 h. After cooling to room temperature naturally, the solids precipitate in the bottom of the autoclave was filtrated and washed with water several times to dissolve other impurities. The crystals of the title complex were recrystallized from the water solution after four days.

Experimental details

All hydrogen atoms were geometrically placed in idealized positions and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Comment

Coordination polymers of metal ions linked by organic ligands have essential applications, such as gas storage, gas and liquid separation, water purification, catalytic, and drug delivery. Therefore, the design and controlled synthesis of coordination polymers have attracted increasing attention [4, 5]. 5-Ethoxisophtalic acid has versatile coordination modes and is a widely used polydentate ligand in coordination synthesis [6, 7]. In addition, the two N atoms of 2,2'-bipyridine are easy to coordinate to metal ions, which often provide a stable framework, and have been widely employed in the construction of coordination polymers, too [8, 9]. The dual ligand coordination polymers isophtalic acid and bipyridine have received extensive attention [10–14]. This paper provided a novel crystal structure constructed by 5-ethoxisophtalic acid and 2,2'-bipyridine as bridging and chelating ligands respectively.

In the title compound crystal structure, as shown in the figure, the Cd center 7-coordinated by four oxygen atoms from two 5-ethoxisophtalate ligands, two nitrogen atoms from the 2,2'-bipyridine ligand, and one oxygen atom from the water molecule. The five atoms (O2, O3, O4, O5, and N1)

in the equatorial plane around the Cd form a highly distorted planar arrangement, and the N atom of 2,2'-bipyridine (N2) and the O atom of the water molecule (O6) are in the axial positions. The 5-ethoxyisophthalate ligands link neighboring Cd atoms forming a one-dimensional helical chain decorated with 2,2'-bipyridine ligands. The spiral chain is further extended into a three-dimensional supramolecular structure *via* intermolecular π - π interactions, similar to reported configurations [11, 13, 14].

Interaction analysis shows that the neighboring 5-ethoxyisophthalate ligands and water molecules create three hydrogen bonds (O6-H6A...O7, O6-H6B...O4, and O7-H7D...O3). Their bond lengths are 1.915(3), 1.871(3), and 1.916(3) Å, and their angles are 144.69(16)°, 167.62(15)°, and 165.2(2)°, respectively. These strong hydrogen bonds link the molecules and stabilize the water molecules in the material.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was financially supported by the Key Scientific Research Projects of Colleges and Universities in Henan Province (22A430032).

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

References

- Agilent Technologies. *CrysAlis PRO Software System, Version 171.38.41r*; Agilent Technologies UK Ltd: Oxford, UK, 2015.
- Dolomanov O. V., Bourhis L. J., Gildea R. J., Howard J. A. K., Puschmann H. OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* 2009, 42, 339–341.
- Bourhis L. J., Dolomanov O. V., Gildea R. J., Howard J. A. K., Puschmann H. The anatomy of a comprehensive constrained, restrained refinement program for the modern computing environment—Olex2 dissected. *Acta Crystallogr.* 2015, A71, 59–75.
- Kuznetsova A., Matveevskaya V., Pavlov D., Yakunenko A., Potapov A. Coordination polymers based on highly emissive ligands: synthesis and functional properties. *Materials* 2020, 13, 2699.
- Emerson A. J., Chahine A., Batten S. R., Turner D. R. Synthetic approaches for the incorporation of free amine functionalities in porous coordination polymers for enhanced CO₂ sorption. *Coord. Chem. Rev.* 2018, 365, 1–22.
- Feng Z., Cheng L., Fan H., Liu J., Han F. Two new Co (II)/Zn (II) coordination polymers: photocatalytic and luminescent property, and application value on alveolar bone reconstruction. *J. Mol. Struct.* 2022, 1250, 131794.
- Hu H., Zhu J., Yang F., Chen Z., Deng M., Weng L., Ling Y., Zhou Y. A robust etb-type metal-organic framework showing polarity-exclusive adsorption of acetone over methanol for their azeotropic mixture. *Chem. Commun.* 2019, 55, 6495–6498.
- Yang H., Duan X.-Y., Lai J.-J., Zhao Y., Wang X.-J., Wei M.-L. Proton-conductive cocrystals of twin isomers of coordination polymers in situ formed by keggian anions and Cu (II)-4, 4'-bis (hydroxymethyl)-2, 2'-bipyridine complex moieties. *Inorg. Chem.* 2018, 58, 446–455.
- Mautner F. A., Fischer R. C., Reichmann K., Gullett E., Ashkar K., Massoud S. S. Synthesis and characterization of 1D and 2D cadmium (II)-2,2-bipyridine-*N, N'*-dioxide coordination polymers bridged by pseudohalides. *J. Mol. Struct.* 2019, 1175, 797–803.
- Shen S.-M. *catena*-poly [[aqua (2, 2'-bipyridine) manganese (II)]- μ -5-methoxyisophthalato- κ^3O, O'] monohydrate]. *Acta Crystallogr.* 2009, E65, m1173.
- Husain A., Parveen R., Dastidar P. New series of ZnII/CdII mixed ligand coordination polymers: toward the design of metallogels. *Cryst. Growth Des.* 2015, 15, 5075–5085.
- Nartey K. A., Wang X., Zhang J., Hu J. A thermal stable fluorescent coordination polymer for sensing nitro aromatic compounds and metal ions with high selectivity and sensitivity in aqueous media. *Opt. Mater.* 2021, 119, 111327.
- Lin Y., Zhang X., Chen W., Shi W., Cheng P. Three cadmium coordination polymers with carboxylate and pyridine mixed ligands: luminescent sensors for FeIII and CrVI ions in an aqueous medium. *Inorg. Chem.* 2017, 56, 11768–11778.
- He Y.-C., Xiao L.-Y., Yuan Z.-H., Zhang J., Wang Y., Xu N. Two coordination polymers constructed by 5-[(4-carboxyphenoxy) methyl] benzene-1, 3-dicarboxylic acid and 2, 2'-bipyridine: syntheses, structures and luminescence properties. *Acta Crystallogr.* 2019, C75, 1562–1568.