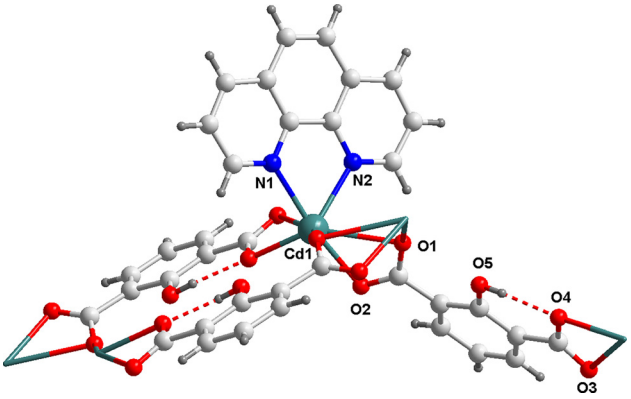
Qiang Zhao, Xinyu Chen and Guang Huang*

The crystal structure of *catena*-poly [(1,10-phenanthroline-κ^2*N*,*N*′)-(μ$_3$-2-hydroxybenzene-1,3-dicarboxylato-κ^5*O*,*O*′:*O*″,*O*‴:*O*‴)cadmium(II)], $C_{20}H_{12}CdN_2O_5$



https://doi.org/10.1515/ncrs-2022-0389
Received July 28, 2022; accepted September 13, 2022; published online October 3, 2022

Abstract

$C_{20}H_{12}CdN_2O_5$, monoclinic, *P*2/*c* (no. 13), *a* = 8.6308(6) Å, *b* = 10.5180(6) Å, *c* = 18.6325(11) Å, *β* = 102.210(6)°, *V* = 1653.17(18) Å^3, *Z* = 4, $R_{gt}(F)$ = 0.0374, $wR_{ref}(F^2)$ = 0.0856, *T* = 250 K.

CCDC no.: 2206932

A part of the polymeric title 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 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.13 × 0.12 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.36 mm^{-1}
Diffractometer, scan mode:	SuperNova, *ω*
θ_{max}, completeness:	29.5°, >99%
$N\,(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8061, 3904, 0.029
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3151
$N(param)_{refined}$:	254
Programs:	CrysAlis^{PRO} [1], Olex2 [2], SHELX [3, 4]

Source of material

A mixture of 0.10 mmol $Cd(NO_3)_2{\cdot}4H_2O$ (0.0198 g), 0.10 mmol 2-hydroxybenzene-1,3-dicarboxylic acid (0.0166 g), 0.10 mmol 1,10-phenanthroline monohydrate (0.0198 g), 0.20 mmol NaOH (0.008 g) and 10 mL water was transferred to a 25 mL teflon-lined reactor, heated at 433 K in an oven for 72 h, then cooled to room temperature. The crystals were washed by deionized water and dried, yield 65% (based on 2-hydroxybenzene-1,3-dicarboxylic acid).

Experimental details

The structure was solved by direct methods with the SHELXS-2018 program. All H-atoms from C atoms were positioned with idealized geometry and refined isotropically (U_{iso} (H) = 1.2 U_{eq} (C)) using a riding model with C—H = 0.930 Å. The H-atoms from O atoms positioned with Q peaks refined isotropically with the distance of O—H = 0.820 Å (U_{iso} (H) = 1.5U_{eq} (O)). The largest difference electron density peak of about 1.3 e/Å^3 near C6 suggests that a small amount of a different carboxylate ligand is part of the measured crystal.

Comment

Cadmium(II) cation co-coordinate with aromatic polycarboxylic acids and multi-pyridine ligands to construct

***Corresponding author: Guang Huang**, Sino-Portugal Belt and Road Joint Laboratory on Science of Cultural Heritage Conservation, City University of Macau, Macau, P. R. China; and Southern Marine Science and Engineering Guangdong Laboratory (Zhuhai), Zhuhai, Guangdong, P. R. China, E-mail: ghuang@cityu.mo. https://orcid.org/0000-0002-9171-3641

Qiang Zhao and Xinyu Chen, Sino-Portugal Belt and Road Joint Laboratory on Science of Cultural Heritage Conservation, City University of Macau, Macau, P. R. China

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

Atom	x	y	z	U_{iso}^*/U_{eq}
C1	0.3695 (4)	0.8365 (4)	0.0515 (2)	0.0382 (9)
H1	0.336367	0.756610	0.033028	0.046*
C2	0.2952 (5)	0.9432 (4)	0.0163 (2)	0.0479 (10)
H2	0.213366	0.934631	−0.024842	0.057*
C3	0.3430 (5)	1.0594 (4)	0.0423 (2)	0.0518 (12)
H3	0.293761	1.131426	0.018957	0.062*
C4	0.4657 (5)	1.0728 (3)	0.1039 (2)	0.0409 (9)
C5	0.5258 (6)	1.1913 (4)	0.1345 (3)	0.0580 (13)
H5	0.482046	1.266223	0.112630	0.070*
C6	0.6437 (6)	1.1976 (4)	0.1940 (3)	0.0631 (14)
H6	0.680347	1.276881	0.212146	0.076*
C7	0.7152 (5)	1.0853 (4)	0.2305 (2)	0.0466 (10)
C8	0.8364 (6)	1.0861 (5)	0.2936 (3)	0.0664 (14)
H8	0.874725	1.162858	0.314995	0.080*
C9	0.8980 (5)	0.9745 (5)	0.3235 (2)	0.0602 (13)
H9	0.979313	0.974455	0.365188	0.072*
C10	0.8393 (4)	0.8617 (4)	0.2917 (2)	0.0452 (10)
H10	0.882375	0.785986	0.312726	0.054*
C11	0.6602 (4)	0.9662 (3)	0.2014 (2)	0.0316 (8)
C12	0.5346 (4)	0.9601 (3)	0.13741 (19)	0.0299 (7)
C13	0.7312 (4)	0.5388 (3)	0.30331 (18)	0.0304 (7)
C14	0.8224 (4)	0.4643 (3)	0.36670 (17)	0.0243 (7)
C15	0.9675 (4)	0.4129 (3)	0.36177 (18)	0.0283 (7)
H15	1.005965	0.426092	0.319349	0.034*
C16	1.0564 (4)	0.3426 (3)	0.4182 (2)	0.0312 (8)
H16	1.154504	0.310394	0.414228	0.037*
C17	0.9991 (4)	0.3207 (3)	0.48016 (18)	0.0274 (7)
H17	1.058028	0.271093	0.517487	0.033*
C18	0.8547 (3)	0.3708 (3)	0.48870 (17)	0.0230 (6)
C19	0.7669 (3)	0.4450 (3)	0.43195 (17)	0.0247 (7)
C20	0.7964 (4)	0.3452 (3)	0.55748 (18)	0.0281 (7)
Cd1	0.61778 (3)	0.66810 (2)	0.17074 (2)	0.03047 (10)
N1	0.4861 (3)	0.8441 (2)	0.11056 (15)	0.0275 (6)
N2	0.7232 (3)	0.8564 (3)	0.23188 (16)	0.0327 (7)
O1	0.6350 (3)	0.6223 (2)	0.31404 (14)	0.0409 (6)
O2	0.7604 (3)	0.5175 (2)	0.24138 (13)	0.0440 (7)
O3	0.8661 (3)	0.2708 (3)	0.60399 (13)	0.0446 (6)
O4	0.6687 (3)	0.4017 (2)	0.56374 (13)	0.0395 (6)
O5	0.6271 (3)	0.4971 (2)	0.43793 (14)	0.0411 (6)
H5A	0.601361	0.469848	0.474973	0.062*

various complexes. Thanks to the modifiability of aromatic rings, many cadmium(II) complexes based on benzene-1,3-dicarboxylate or its derivatives have been reported [5–18].

The asymmetric unit is made of one Cd(II) cation, one 2-hydroxybenzene-1,3-dicarboxylate anion and one 1, 10-phenanthroline molecule. The Cd(II) is seven-coordinated: two nitrogen atoms from one 1,10-phenanthroline, five oxygen atoms from three 2-hydroxybenzene-1,3-dicarboxylate anions. The adjacent two Mn(II) cations are linked by two 2-hydroxybenzene-1,3-dicarboxylate anions to generate 1D structure, further forming a 3D supramolecular structure by π···π weak interaction of 1,10-phenanthroline molecules. The Cd–N distances are 2.332 and 2.368 Å, and the Cd–O distances are 2.253, 2.310, 2.252, 2.253, 2.676 and 2.771 Å, respectively, which are similar to the reported results [5–16].

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

Research funding: This study was funded by the Macau Science and Technology Development Fund (FDCT-0039/2021/ITP).

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

References

1. CrysAlis^{Pro} 1.171.38.43f. Rigaku OD: Oxfordshire, England, 2015.
2. 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.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Sheldrick G. Using phases to determine the space group. *Acta Crystallogr.* 2018, *A74*, a353.
5. Zhang L.-Y., Liu G.-F., Zheng S.-L., Ye B.-H., Zhang X.-M., Chen X.-M. Helical ribbons of cadmium(II) and zinc(II) dicarboxylates with bipyridyl-like chelates-syntheses, crystal structures and photoluminescence. *Eur. J. Inorg. Chem.* 2003, *2003*, 2965–2971.
6. Ren H., Yu L.-X., Zhang L.-R., Chen Y.-L., Jiang J.-H., Zhang P., Song T-.Y. Synthesis, structures and properties of two carboxylate-bridged coordination polymers with linear and ladder chain motifs. *J. Coord. Chem.* 2008, *61*, 3960–3972.
7. Li X., Wu B.-L., Liu W., Niu C.-Y., Niu Y.-Y., Zhang H.-Y. Syntheses, crystal structures, and magnetic properties of five new coordination compounds bearing ferrocenedicarboxylate ligands. *J. Coord. Chem.* 2009, *62*, 3142–3156.
8. Zhang K.-L., Chang Y., Zhang J.-B., Yuan L.-M., Deng Y., Diao G.-W., Ng S. W. Synthesis, crystal structures and characterization of four coordination polymers based on 5-amino-2,4,6-triiodoisophthalic acid. *J. Solid State Chem.* 2011, *184*, 1263–1272.
9. Zang S.-Q., Fan Y.-J., Li J.-B., Hou H.-W., Mak T. C. W. Halogen bonding in the assembly of coordination polymers based on 5-iodo-isophthalic acid. *Cryst. Growth Des.* 2011, *11*, 3395–3405.
10. Xu Y.-M., Zhao X.-L., Hu T.-P. Synthesis, structure and thermal stability of a cadmium coordination polymer: $[Cd(phen)(4,6\text{-dimethyl-5-nitro-isophthalic acid})]_n$. *Chin. J. Struct. Chem.* 2011, *30*, 1442–1446.
11. Kariem M., Yawer M., Sheikh H. N. Solvent induced synthesis, structure and properties of coordination polymers based on 5-hydroxyisophthalic acid as linker and 1,10-phenanthroline as auxiliary ligand. *J. Solid State Chem.* 2015, *231*, 239–247.

12. Wang J., Lin P., Du S.-W. Temperature dependent assembly, structural diversity and luminescent property in two novel Cd coordination polymers. *Chin. J. Struct. Chem.* 2015, *34*, 945–952.
13. Zhang D.-W., Zhao G.-Y. Synthesis, crystal structure, and photoluminescent property of a new 1D→2D interdigitated framework. *Synth. React. Inorg. Met.-Org. Nano-Met. Chem.* 2015, *45*, 524–526.
14. Wu D., Huang W., Xu J., Kirillov A., Jiang J. Tuning topological and dimensional versatility from 1D to 3D of Zn/Cd luminescent biphenyl-3,5-dicarboxylate coordination polymers by ancillary ligand. *J. Coord. Chem.* 2016, *69*, 2200–2209.
15. Yin W.-Y., Zhuang G.-Y., Huang Z.-L., Cheng H.-J., Zhou L., Ma M.-H., Wang H., Tang X.-Y., Ma Y.-S., Yuan R.-X. Structural diversification and photocatalytic properties of three Cd(II) coordination polymers decorated with different auxiliary ligands. *J. Solid State Chem.* 2016, *235*, 93–99.
16. Song W., Xu X.-L., Yang Y.-J., Li S. H. Crystal structure of catena-poly[(μ_3-5-carboxy-2-(pyridin-4-yl)benzoato-$\kappa^5O,O':O'',O''':O'''$)(1,10-phenanthroline-$\kappa^2N,N'$)cadmium(II)], $C_{100}H_{60}N_{12}O_{16}Cd_4$. *Z. Kristallogr. N. Cryst. Struct.* 2018, *233*, 755–756.
17. Shi Q.-Y., Li Z.-C., Cheng Z.-S., Tan J.-B., Liu J.-L. Bis(μ-2-hydroxybenozato)-$\kappa^3O,O':O';\kappa^3O:O,O'$-bis[(2-hydroxybenozato-$\kappa^2O,O'$)(1,10-phenanthroline-$\kappa^2N,N'$)cadmium(II)]. *Acta Crystallogr.* 2008, *E64*, m1458.
18. Banrabah R., Viossat B., Lemoine P. Crystal structure of bis(1,10-phenanthroline-κ^2N,N')bis(2-hydroxybenzoato-κO^1; $\kappa^2O^1,O^{1'}$)cadmium(II) – methanol (1:1), $Cd(C_{12}H_8N_2)_2(C_7H_5O_3)_2 \cdot CH_3OH_2$. *Z. Kristallogr. N. Cryst. Struct.* 2011, *226*, 291–293.