Peng-Jiang Li, Shi-Jun Chen, Zhi-Bin Yu and Shi-Hui Li*

Crystal structure of *cyclo*[diaqua-bis(μ_2 -3',5-dicarboxy-[1,1'-biphenyl]-3,4'-dicarboxylato- $\kappa^4 O, O': O'', O'''$)-bis(4,4'-bis(pyrid-4-yl)biphenyl-K¹N) dicadmium(II)], $C_{76}H_{52}Cd_2N_4O_{18}$

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:	0.43 × 0.35 × 0.21 mm
Wavelength:	0.71073 Å
μ :	0.77 mm ⁻¹
Diffractometer, scan mode:	Xcalibur, omega
$\theta_{\max}$, completeness:	25.5°, 98%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	14246, 0.049, 0.0487
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4625
$N(\text{param})_{\text{refined}}$:	454
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3]

Source of material

A mixture of $Cd(NO_3)_2 \cdot 2H_2O$ (0.1 mmol, 0.027 g), 4,4'-bis(pyrid-4-yl)biphenyl (0.06 g, 0.2 mmol) and 3,3',4,5'-biphenyl tetracarboxylic acid (H4btc) (0.1 mmol, 0.033 g), was dissolved in 8 mL H_2O . The solution was heated in a 23 mL Teflon-lined autoclave under autogenous pressure at 393 K for five days. Cooling to room temperature gave colorless crystals.

Experimental details

Coordinates of hydrogen atoms were added using riding models [3].

Comment

Mixed ligand-MOFs can be constructed from metal ions, carboxylic acids and nitrogen-containing ligands. Normally N-containing ligands are electrically neutral, while carboxylate ligands are negatively charged. Using organic aromatic multicarboxylates yield networks and have been widely utilized to construct MOFs [4, 5]. On the other hand, 4,4'-bis(pyrid-4-yl)biphenyl often serves as bridging ligand [6].

In the title complex, there are one Cd(II) ion, one coordinated water molecule, one H_2btc^{2-} ligand, and one 4,4'-bis(pyrid-4-yl)biphenyl ligand in the asymmetric unit. Cd1 is six-coordinated with a distorted octahedral geometry by four O atoms from two H_2btc^{2-} and one coordinated water molecule, and one N atom from the biphenyl derivate (see the figure).

<https://doi.org/10.1515/ncrs-2020-0192>

Received April 20, 2020; accepted June 19, 2020; available online July 2, 2020

Abstract

$C_{76}H_{52}Cd_2N_4O_{18}$, triclinic, $P\bar{1}$ (no. 2), $a = 8.5834(4)$ Å, $b = 10.7034(5)$ Å, $c = 17.6105(8)$ Å, $\alpha = 99.328(4)^\circ$, $\beta = 100.580(4)^\circ$, $\gamma = 95.281(4)^\circ$, $V = 1556.94(13)$ Å³, $Z = 1$, $R_{\text{gt}}(F) = 0.0477$, $wR_{\text{ref}}(F^2) = 0.1183$, $T = 293(2)$ K.

CCDC no.: 2010900

*Corresponding author: Shi-Hui Li, College of Chemistry and Chemical Engineering, Luoyang Normal University, Luoyang, Henan 471934, P.R. China, e-mail: shihui471022@126.com. <https://orcid.org/0000-0002-2227-7903>

Peng-Jiang Li and Zhi-Bin Yu: Changbei Operating Company, Changqing Oilfield Co. of Petro China, Xi'an 710000, Shaanxi Province, P.R. China

Shi-Jun Chen: College of Chemistry and Chemical Engineering, Xi'an SHIYOU University, Xi'an 710065, Shaanxi Province, P.R. China

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}
Cd1	0.71513(3)	0.37584(3)	0.916003(16)	0.02833(13)
N1	0.8222(5)	0.4814(3)	0.8310(2)	0.0374(9)
N2	1.7300(5)	1.4723(4)	0.4479(2)	0.0485(11)
O1	0.7512(4)	0.5485(3)	1.00903(19)	0.0460(8)
H1A	0.8447	0.5871	1.0142	0.069*
H1B	0.7501	0.5257	1.0530	0.069*
O2	0.4412(4)	0.3764(3)	0.89484(18)	0.0440(8)
O3	0.5368(3)	0.2383(3)	0.81684(17)	0.0389(8)
O4	0.3557(4)	0.4143(3)	0.71646(19)	0.0468(8)
O5	0.1430(4)	0.3476(4)	0.6199(2)	0.0554(10)
H5	0.1844	0.4023	0.5997	0.083*
O6	−0.5251(4)	−0.0428(3)	0.8826(2)	0.0557(10)
O7	−0.4700(4)	−0.1838(4)	0.9572(3)	0.0748(14)
H7	−0.5648	−0.1844	0.9592	0.112*
O8	0.0422(3)	−0.3095(3)	1.03363(16)	0.0307(7)
O9	0.2496(3)	−0.2307(3)	0.99374(18)	0.0361(7)
C1	0.7414(6)	0.5540(5)	0.7870(3)	0.0550(14)
H1	0.6322	0.5523	0.7853	0.066*
C2	0.8113(6)	0.6308(5)	0.7443(3)	0.0552(14)
H2	0.7492	0.6792	0.7145	0.066*
C3	0.9734(5)	0.6370(4)	0.7452(2)	0.0344(10)
C4	1.0542(6)	0.5605(5)	0.7907(3)	0.0467(13)
H4	1.1633	0.5597	0.7934	0.056*
C5	0.9775(6)	0.4867(5)	0.8314(3)	0.0463(13)
H5A	1.0369	0.4369	0.8614	0.056*
C6	1.0575(6)	0.7240(4)	0.7050(3)	0.0379(11)
C7	0.9854(6)	0.7572(5)	0.6352(3)	0.0467(12)
H7A	0.8811	0.7221	0.6120	0.056*
C8	1.0666(6)	0.8424(5)	0.5991(3)	0.0443(12)
H8	1.0166	0.8611	0.5515	0.053*
C9	1.2196(6)	0.8992(4)	0.6331(2)	0.0358(10)
C10	1.2903(6)	0.8640(5)	0.7020(3)	0.0517(13)
H10	1.3944	0.8994	0.7256	0.062*
C11	1.2125(6)	0.7786(5)	0.7372(3)	0.0474(13)
H11	1.2652	0.7571	0.7834	0.057*
C12	1.3052(6)	0.9960(4)	0.5993(2)	0.0356(11)
C13	1.2661(6)	1.0068(4)	0.5199(3)	0.0401(11)
H13	1.1833	0.9510	0.4863	0.048*
C14	1.3491(6)	1.0993(4)	0.4910(3)	0.0392(11)
H14	1.3219	1.1032	0.4379	0.047*
C15	1.4718(5)	1.1866(4)	0.5390(3)	0.0347(10)
C16	1.5089(6)	1.1760(4)	0.6179(3)	0.0404(11)
H16	1.5894	1.2334	0.6520	0.048*
C17	1.4292(6)	1.0829(4)	0.6465(3)	0.0418(11)
H17	1.4592	1.0777	0.6993	0.050*
C18	1.5617(6)	1.2850(4)	0.5079(3)	0.0376(11)
C19	1.5021(7)	1.3212(6)	0.4379(3)	0.0620(16)
H19	1.4027	1.2833	0.4087	0.074*
C20	1.5886(7)	1.4131(6)	0.4109(3)	0.0621(16)
H20	1.5442	1.4349	0.3633	0.074*
C21	1.7885(7)	1.4370(5)	0.5153(3)	0.0553(14)
H21	1.8892	1.4753	0.5426	0.066*
C22	1.7093(6)	1.3470(5)	0.5473(3)	0.0503(13)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H22	1.7556	1.3282	0.5955	0.060*
C23	0.4211(5)	0.2845(4)	0.8371(2)	0.0269(9)
C24	0.2544(5)	0.2257(4)	0.8007(2)	0.0264(9)
C25	0.1868(5)	0.1400(4)	0.8399(2)	0.0295(10)
H25	0.2472	0.1227	0.8856	0.035*
C26	0.0313(5)	0.0784(4)	0.8132(2)	0.0262(9)
C27	−0.0556(5)	0.1027(4)	0.7433(2)	0.0346(10)
H27	−0.1591	0.0622	0.7235	0.042*
C28	0.0126(5)	0.1870(4)	0.7037(3)	0.0386(11)
H28	−0.0468	0.2027	0.6572	0.046*
C29	0.1652(5)	0.2489(4)	0.7306(2)	0.0304(10)
C30	−0.0378(5)	−0.0106(4)	0.8585(2)	0.0262(9)
C31	0.0598(5)	−0.0843(4)	0.9007(2)	0.0261(9)
H31	0.1676	−0.0797	0.8986	0.031*
C32	−0.0017(5)	−0.1635(4)	0.9454(2)	0.0235(9)
C33	−0.1617(5)	−0.1718(4)	0.9478(2)	0.0259(9)
H33	−0.2037	−0.2267	0.9769	0.031*
C34	−0.2615(5)	−0.0985(4)	0.9068(2)	0.0275(9)
C35	−0.1988(5)	−0.0184(4)	0.8627(2)	0.0276(9)
H35	−0.2648	0.0308	0.8355	0.033*
C36	0.2330(6)	0.3448(4)	0.6888(3)	0.0358(10)
C37	0.1030(5)	−0.2395(4)	0.9931(2)	0.0268(9)
C38	−0.4319(5)	−0.1036(4)	0.9132(2)	0.0326(10)

The Cd–N bond length is 2.284(3) Å and the Cd–O ones are in the range of 2.309(3) to 2.396(2) Å. The bond angles of O–Cd–O are in the range of 56.12(8)° to 155.66(10)°. The coordination leads to a dinuclear cyclic dimer.

References

- Agilent Technologies: CrysAlis^{PRO} Software system, version 1.171.35.15. Agilent Technologies UK Ltd, Oxford, UK (2011).
- Sheldrick, G. M.: SHELXT – Integrated space-group and crystal-structure determination. *Acta Crystallogr. A* **71** (2015) 3–8.
- Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Crystallogr. C* **71** (2015) 3–8.
- Li, S. H.; Zhao, Y.: Synthesis and crystal structure of a new three-dimensional Co(II) coordination polymer based on 3,3', 4,5'-biphenyl tetracarboxylic acid and 4,4'-bipyridine. *Inorg. Nano-Met. Chem.* **47** (2017) 256–259.
- Li, S. H.; Zang, J.; Guo, J. B.: Synthesis and crystal structure of a new three-dimensional Ho(III) coordination polymer based on 5'-(4-carboxyphenyl)-[1,1':3',1''-terphenyl]-4,4''-dicarboxylic acid. *Inorg. Nano-Met. Chem.* **12** (2017) 1746–1749.
- Li, S. H.; Hu, X. Z.; Zhao, Y.: Crystal structure of catena poly [diaqua-μ₂-4,4'-biphenyl-4,4'-diylidipyridine-κ²N:N'-bis (5-carboxy-2,6-dimethylpyridine-3-carboxylato-*O*)nickel(II)] dihydrate. *Z. Kristallogr. NCS* **231** (2016) 157–159.