Guang-Zhen Liu*, Lu-Ping Ma and Ru Jia

Crystal structure of the coordination polymer *catena*-poly[(1,2-di(pyridin-4-yl)ethane- κN)-(μ_2 -2-nitroisophthalato- $\kappa^2 O:O'$)zinc(II)], $C_{20}H_{17}N_3O_7Zn$

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.36 × 0.33 × 0.27 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.28 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
$\theta_{\max}$, completeness:	28.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	20317, 4466, 0.038
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3729
$N(\text{param})_{\text{refined}}$:	281
Programs:	Olex2 [1], SHELX [2, 3], CrysAlis ^{PRO} [4]

<https://doi.org/10.1515/ncrs-2019-0510>

Received July 18, 2019; accepted September 13, 2019; available online October 9, 2019

Abstract

$C_{20}H_{17}N_3O_7Zn$, monoclinic, $P2_1/n$ (no. 14), $a = 10.4368(3)$ Å, $b = 15.1527(5)$ Å, $c = 12.9007(3)$ Å, $\beta = 101.900(3)^\circ$, $V = 1996.36(10)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0410$, $wR_{\text{ref}}(F^2) = 0.0898$, $T = 293(2)$ K.

CCDC no.: 1953494

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.

Source of material

All reagents used in the synthesis were of analytical grade and used as purchased without further purification. The mixtures of 2-nitroisophthalic acid (nipc) (0.1 mmol, 21.1 mg), $Zn(OAc)_2 \cdot 2H_2O$ (0.1 mmol, 22.0 mg), 1,2-bis(4-pyridyl)ethane (bpa) (0.1 mmol, 18.4 mg) and H_2O (6.0 mL) was placed in a 23 mL Teflon liner stainless steel reactor. The vessel was

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Zn1	0.89979(3)	0.38717(2)	0.56375(2)	0.02860(10)
O1	0.68499(19)	0.35737(14)	0.69036(15)	0.0485(5)
O1W	0.94251(19)	0.49672(12)	0.64465(13)	0.0412(5)
H1WA	0.9667	0.5373	0.6053	0.062*
H1WB	1.0114	0.4891	0.6944	0.062*
O2	0.8708(2)	0.29882(13)	0.66243(15)	0.0465(5)
O3	0.53519(17)	0.14114(12)	0.98166(13)	0.0347(4)
O4	0.6547(2)	0.19044(16)	1.13325(14)	0.0574(6)
O5	0.5052(2)	0.30051(15)	0.85146(19)	0.0579(6)
O6	0.5387(2)	0.19727(15)	0.74532(15)	0.0556(6)
N1	0.7530(2)	0.41113(14)	0.43740(16)	0.0322(5)
N2	0.1051(3)	0.48538(18)	−0.16684(18)	0.0522(7)
N3	0.5746(2)	0.24749(15)	0.81933(17)	0.0342(5)
C1	0.7818(3)	0.31110(16)	0.71605(19)	0.0312(6)
C2	0.8100(2)	0.26564(16)	0.82256(18)	0.0268(5)
C3	0.9390(2)	0.25657(18)	0.8762(2)	0.0382(6)
H3	1.0065	0.2699	0.8416	0.046*
C4	0.9687(3)	0.2281(2)	0.9800(2)	0.0458(7)
H4	1.0556	0.2233	1.0152	0.055*
C5	0.8700(3)	0.20672(18)	1.0313(2)	0.0384(6)
H5	0.8909	0.1896	1.1020	0.046*
C6	0.7393(2)	0.21026(16)	0.97938(18)	0.0281(5)
C7	0.7129(2)	0.24070(15)	0.87552(17)	0.0249(5)
C8	0.6368(3)	0.18000(17)	1.03781(19)	0.0323(6)
C9	0.6605(3)	0.4719(2)	0.4366(2)	0.0462(7)
H9	0.6587	0.5023	0.4989	0.055*
C10	0.5673(3)	0.4917(2)	0.3470(3)	0.0573(9)

*Corresponding author: Guang-Zhen Liu, College of Chemistry and Chemical Engineering, and Henan Key Laboratory of Function-Oriented Porous Materials, LuoYang Normal University, Luoyang, Henan 471934, P.R. China, e-mail: gzliuyl@126.com

Lu-Ping Ma and Ru Jia: College of Chemistry and Chemical Engineering, LuoYang Normal University, Luoyang, Henan 471934, P.R. China

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H10	0.5041	0.5345	0.3497	0.069*
C11	0.5683(3)	0.4478(2)	0.2538(2)	0.0526(8)
C12	0.6595(3)	0.3822(2)	0.2559(2)	0.0519(8)
H12	0.6601	0.3488	0.1956	0.062*
C13	0.7498(3)	0.36622(19)	0.3474(2)	0.0413(7)
H13	0.8116	0.3221	0.3470	0.050*
C14	0.4741(4)	0.4708(3)	0.1501(3)	0.0744(12)
H14A	0.4699	0.5345	0.1424	0.089*
H14B	0.5084	0.4471	0.0914	0.089*
C15	0.3409(3)	0.4364(2)	0.1443(2)	0.0573(9)
H15A	0.3018	0.4649	0.1975	0.069*
H15B	0.3448	0.3735	0.1584	0.069*
C16	0.2575(3)	0.4536(2)	0.0346(2)	0.0467(7)
C17	0.2602(3)	0.3977(2)	−0.0491(2)	0.0525(8)
H17	0.3136	0.3480	−0.0394	0.063*
C18	0.1835(3)	0.4155(2)	−0.1475(2)	0.0533(8)
H18	0.1868	0.3768	−0.2029	0.064*
C19	0.1050(4)	0.5395(2)	−0.0874(3)	0.0664(10)
H19	0.0516	0.5892	−0.0995	0.080*
C20	0.1792(4)	0.5269(2)	0.0126(3)	0.0679(11)
H20	0.1765	0.5682	0.0655	0.081*

heated to 393 K for 4 days, and then slowly cooled to room temperature. Colorless crystals were obtained, and further crystals were filtered off, washed with mother liquid, and dried under ambient conditions. Yield 46%.

Experimental details

A suitable crystal was selected and mounted on a SuperNova, Single source at offset/far, EosS2 diffractometer. Using Olex2 [1], the structure was solved with the ShelXT [2] structure solution program and refined with the ShelXL [3] refinement package. All hydrogen atoms were placed in calculated positions with a riding model.

Comment

A coordination polymer is generally formed by self-assembly through coordination bonds between ligands and central metal ions or clusters to produce one-, two-, or three-dimensional metal-organic coordination network [5–7]. Porous coordination polymers (PCPs) are of great interest due to their potential applications in guest exchange and separation, gas storage, and selective catalysis, etc [8–10]. For many years, it has been demonstrated that intermolecular weak interactions including H-bonds, π – π interactions, and even van der Waals forces are able to create stable molecular networks in crystalline solids that are reproducible, thermally stable, and resilient upon undergoing dynamic processes [11, 12]. Our work focuses on the design and synthesis of functional PCPs based on multitopic organic ligands.

We select 2-nitroisophthalate (nipc) as the assembly ligand, considering its following characteristics: (a) it is a bent ligand and with the two carboxylate groups located at appropriate angles (approximately 120°), which may allow it to connect metal ions to generate 1D helical chains; (b) the nitro and carboxylate groups of the ligand can act as hydrogen-bond donors. In addition, the dipyrindyl-type pillar ligands, such as 4,4'-bipyridine (bpy), 1,2-bis(4-pyridyl)ethane (bpa), 1,2-di(4-pyridyl)-ethylene(dpe), 1,3-di(4-pyridyl)propane (bpp), and *trans*-1-(2-pyridyl)-2-(4-pyridyl)ethylene (ppe), are known to be ideal connectors for the propagation of coordination networks and can provide a synergistic coordination together with carboxylate ligands [13–15]. To the best of our knowledge, coordination polymers constructed from the *nipc* ligand are rather rare.

The asymmetric unit contains one Zn(II) atom, one *bpa* molecule, one *nipc* dianion and one coordinated H₂O. Each Zn(II) ion is four coordinated by three oxygen atoms from two carboxylate groups of *nipc* and one coordinated water (Zn–O: 1.9138(18) Å – 1.9797(17) Å), and one nitrogen atom of the *bpa* ligand (Zn–N: 2.026(2) Å), displaying a highly distorted tetrahedral coordination environment. Each *nipc* anion through a bis(monodentate) coordinated mode bridges adjacent Zn(II) atoms to yield an undulating helical chain along the [111] direction, with *bpa* ligands as free lateral arms; in contrast to a similar 2D network containing analogous ligands [16]. The paralleled chains are connected by H-bonds between H₂O molecule and carboxylate oxygen atom of *nipc* ligand [O(1W)–H(1WA)···O(3); d(O1···O3) = 2.767(2) Å; degree (H–O···O3) = 161.6°] forming an open layered motif. Two adjacent layer blocks are connected by H-bonds between H₂O molecule and pyridine N atoms of *bpa* molecules [O(1W)–H(1WB)···N(2); d(O1···N2) = 2.668(3) Å; degree (H–O···N2) = 155.5°] producing its 3D supramolecular network.

Acknowledgements: This work was supported by the National Natural Science Foundation of China (no. 21571093).

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