Xue-Li Xu*, Shi-Hui Li and Jin-Bo Guo

Crystal structure of poly[diaqua-bis(μ_2 -4-(3-(pyridin-3-yl)-1*H*-1,2,4-triazol-5-yl)benzoato- $\kappa^2 N$:O)nickel(II)], $C_{28}H_{22}O_6N_8Ni$

Table 1: Data collection and handling.

Crystal:	Green block
Size:	0.41 × 0.35 × 0.29 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.77 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
$\theta_{\max}$, completeness:	25.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8205, 2545, 0.017
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2238
$N(\text{param})_{\text{refined}}$:	195
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	0.0000	0.49921(7)	0.2500	0.04562(19)
O2	−0.04065(8)	0.3014(4)	0.14191(16)	0.0719(7)
H2A	−0.0288	0.2202	0.1813	0.108*
H2B	−0.0749	0.3084	0.1414	0.108*
O3	0.06292(7)	0.4870(3)	0.21086(14)	0.0504(5)
O4	0.13115(7)	0.3379(3)	0.33698(12)	0.0458(5)
N1	0.30348(7)	0.3875(3)	0.10851(14)	0.0343(4)
N2	0.28204(8)	0.5221(3)	−0.03601(15)	0.0360(5)
N3	0.23890(8)	0.5386(3)	−0.01489(15)	0.0340(4)
H3	0.2076	0.5930	−0.0514	0.041*
N4	0.45657(8)	0.1890(4)	0.13842(15)	0.0482(6)
C1	0.11146(9)	0.4168(4)	0.25550(17)	0.0384(6)
C2	0.14775(9)	0.4317(3)	0.20658(17)	0.0344(5)
C3	0.13429(10)	0.5476(4)	0.12892(18)	0.0399(6)
H3A	0.1023	0.6192	0.1064	0.048*
C4	0.16781(10)	0.5586(4)	0.08419(18)	0.0381(5)
H4	0.1586	0.6382	0.0325	0.046*
C5	0.21543(9)	0.4503(3)	0.11670(16)	0.0307(5)
C6	0.22889(9)	0.3335(3)	0.19406(16)	0.0364(5)
H6	0.2605	0.2604	0.2159	0.044*
C7	0.19552(9)	0.3251(3)	0.23892(17)	0.0369(5)
H7	0.2051	0.2471	0.2914	0.044*
C8	0.25211(9)	0.4581(3)	0.07106(17)	0.0300(5)
C9	0.31956(9)	0.4313(3)	0.04033(17)	0.0324(5)
C10	0.37389(9)	0.3724(3)	0.04967(17)	0.0364(5)
C11	0.39086(10)	0.4150(4)	−0.01947(19)	0.0429(6)
H11	0.3692	0.4899	−0.0724	0.051*
C12	0.44079(11)	0.3431(4)	−0.0074(2)	0.0519(7)

<https://doi.org/10.1515/ncrs-2020-0250>

Received May 21, 2020; accepted June 24, 2020; available online July 2, 2020

Abstract

$C_{28}H_{22}O_6N_8Ni$, monoclinic, $C2/c$ (no. 15), $a = 27.0100(11)$ Å, $b = 7.4753(5)$ Å, $c = 15.4781(6)$ Å, $\beta = 118.821(3)^\circ$, $V = 2738.0(2)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0404$, $wR_{\text{ref}}(F^2) = 0.1130$, $T = 293(2)$ K.

CCDC no.: 2011922

A part of the title coordination polymer 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

A mixture of $Ni(OAc)_2 \cdot 4 H_2O$ (0.1 mmol, 0.0248 g), THF (10 mL), 4-(3-(pyridin-3-yl)-1*H*-1,2,4-triazol-5-yl)benzoic acid (0.3 mmol, 266 mg) (H4-ptba) was dissolved in 8 mL H_2O .

*Corresponding author: Xue-Li Xu, School of Biological and Chemical Engineering, Nanyang Institute of Technology, Nanyang 473004, Henan Province, P.R. China, e-mail: guojb2020@126.com
Shi-Hui Li and Jin-Bo Guo: College of Chemistry and Chemical Engineering, Luoyang Normal University, Luoyang, Henan 471934, P.R. China. <https://orcid.org/0000-0001-5050-5531> (J.-B. Guo)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H12	0.4534	0.3705	−0.0522	0.062*
C13	0.47185(10)	0.2315(5)	0.07015(19)	0.0530(8)
H13	0.5050	0.1828	0.0759	0.064*
C14	0.40826(10)	0.2618(4)	0.12698(18)	0.0453(7)
H14	0.3973	0.2361	0.1741	0.054*

Then the solution was heated in a 23 mL Teflon-lined autoclave under autogenous pressure at 393 K for five days. After cooling to room temperature green crystals suitable for X-ray diffraction analysis were collected.

Experimental details

Coordinates of hydrogen atoms were introduced based on a riding model. Their *U*_{iso} values were set to 1.2*U*_{eq} of the parent atoms.

Comment

The design and construction of metal-organic frameworks and coordination polymers is still of interest. The organic ligands, which provide various properties in the size, the exhibility, the coordination ability, the number of the *N*-donor ligands etc. have influence on structure of final product [4, 5]. The flexibility of *N*-donor ligands can meet the requirements of the coordination geometry of the metal ions in the assembly process [6–9]. The organic ligand 4-(3-(pyridin-3-yl)-1*H*-1,2,4-triazol-5-yl)benzoate serves as bridging ligand to coordinate to one metal ion.

In the title complex, there are one half of a Ni(II) ion, one 4-ptba ligand, one coordinated water molecule in the asymmetric unit. The nickel atom Ni1 is six-coordinated (located on a twofold axis) forming a distorted octahedral geometry by two N atoms from the 4-ptba ligands (see the figure).

The Ni–N bond length is 2.102(3) Å; and the Ni–O ones are in the range of 2.0631(17) to 2.103(2) Å, respectively. The bond angles of O–Ni–O are in the range of 85.61(8)° to 174.94(12)°.

Acknowledgements: The authors thank the Key Scientific Research Project for Universities of Henan Province (No. 18B150015; No. 20A150028) and Science and Technology Research Project of Henan Province (no. 182102310909) for financial support.

References

1. Agilent Technologies: CrysAlis^{PRO} Software system, version 1.171.35.15. Agilent Technologies UK Ltd, Oxford, UK (2011).
2. Sheldrick, G. M.: SHELXT – Integrated space-group and crystal-structure determination. *Acta Crystallogr.* **A71** (2015) 3–8.
3. Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Crystallogr.* **C71** (2015) 3–8.
4. Li, S. H.; Wu, H. X.; Chai, N.: A cobalt(II) complex based on 5-methoxyisophthalic acid and 1,6-bis(1,2,4-triazol-1-yl)hexane: synthesis, crystal structure and magnetic properties. *Synth. React. Inorg. Met.-Org. Nano-Met. Chem.* **43** (2013) 1487–1491.
5. Zhao, Y.; Zhang, L.: Crystal structure of 4-(3-(pyridin-3-yl)-1*H*-1,2,4-triazol-5-yl)benzoic acid. *Z. Kristallogr. NCS* **231** (2016) 169–170.
6. Song, W.; Li, S. H.; Xu, X. L.: Crystal structure of *catena*-poly[triaqua-(isophthalato-κO)-(μ₂-1,6-di(1*H*-imidazol-1-yl)hexane-κ²N:N')]nickel(II). *Z. Kristallogr. NCS* **233** (2018) 173–175.
7. Zhao, Y.; Wang, K. Q.: Crystal structure of poly[2-1,4-di(1*H*-imidazol-1-yl)butan-κ²N:N'-2-thiophene-2,3-dicarboxylato-κ⁴O, O':O'',O'''nickel(II)]. *Z. Kristallogr. NCS* **228** (2013) 363–364.
8. Li, S. H.; Li, X. L.: A cadmium complex based on 5-methylisophthalic acid and 1,6-bis(imidazol-1-yl)hexane: synthesis, crystal structure, and photoluminescence properties. *Synth. React. Inorg. Met.-Org. Nano-Met. Chem.* **43** (2013) 710–713.
9. Zhao, Y.: Crystal structure of *catena*-poly[diaqua-bis(μ₂-4-((4-(pyridin-2-ylmethoxy)phenyl)diazanyl)benzoato-κ³O,O':N)cadmium(II)], Cd(C₁₉H₁₄O₃N₃)₂(H₂O). *Z. Kristallogr. NCS* **234** (2019) 741–742.