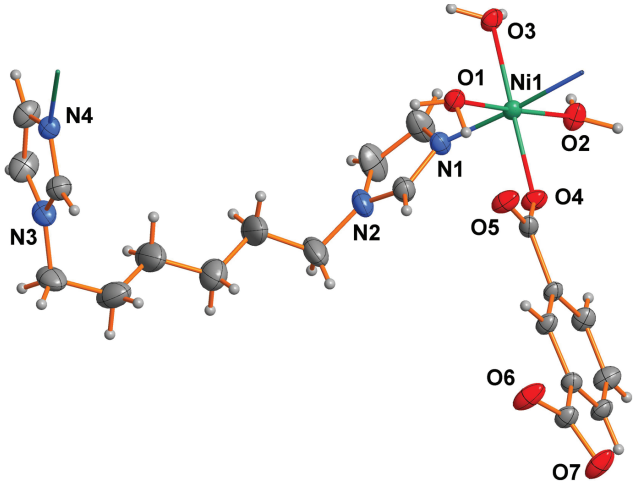
Wei Song*, Shi-Hui Li and Xue-Li Xu

Crystal structure of catena-poly[triaqua-(isophthalato-κ*O*)-(μ$_2$-1,6-di(1*H*-imidazol-1-yl) hexane-κ^2*N*:*N*′)]nickel(II), $C_{20}H_{28}N_4NiO_7$



https://doi.org/10.1515/ncrs-2017-0132
Received October 22, 2017; accepted January 30, 2018; available online February 10, 2018

Abstract

$C_{20}H_{28}N_4NiO_7$, monoclinic, $P2_1/c$ (no. 14), $a = 8.6404(5)$ Å, $b = 21.0392(12)$ Å, $c = 12.2580(6)$ Å, $\beta = 96.839(5)°$, $V = 2212.5(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0476$, $wR_{ref}(F^2) = 0.1092$, $T = 293(2)$ K.

CCDC no.: 1820729

A part of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

A mixture of $Ni(OAc)_2{\cdot}4H_2O$ (0.1 mmol, 0.0248 g), isophthalic acid (0.1 mmol, 0.0167 g), 1,6-di(1*H*-imidazol-1-yl)hexane

***Corresponding author: Wei Song,** School of Biological and Chemical Engineering, Nanyang Institute of Technology, Nanyang 473004, Henan Province, P.R. China, e-mail: luoyangchangchun@126.com
Shi-Hui Li: College of Chemistry and Chemical Engineering, Luoyang Normal University, Luoyang 471022, Henan Province, P.R. China
Xue-Li Xu: School of Biological and Chemical Engineering, Nanyang Institute of Technology, Nanyang 473004, Henan Province, P.R. China

Table 1: Data collection and handling.

Crystal:	Green block
Size:	0.42 × 0.35 × 0.29 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.93 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
θ_{max}, completeness:	25.5°, >98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	11735, 4050, 0.035
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3425
$N(param)_{refined}$:	289
Programs:	SHELX [1, 2]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Ni1	1.05883(4)	0.159707(18)	0.17556(3)	0.02218(14)
O1	1.1346(2)	0.24306(10)	0.26109(16)	0.0297(5)
H1W	1.0851	0.2781	0.2585	0.045*
H2W	1.1283	0.2306	0.3271	0.045*
O2	0.9996(3)	0.07492(10)	0.09390(17)	0.0327(5)
H3W	0.9713	0.0713	0.0275	0.049*
H4W	1.0318	0.0328	0.1177	0.049*
O3	1.1361(2)	0.19373(10)	0.03077(16)	0.0296(5)
H5W	1.1192	0.2361	0.0182	0.044*
H6W	1.0905	0.1714	−0.0306	0.044*
O4	0.9773(3)	0.12337(10)	0.31611(16)	0.0307(5)
O5	1.1009(3)	0.18405(11)	0.44837(17)	0.0357(6)
O6	0.9996(3)	0.14936(11)	0.83490(18)	0.0448(7)
O7	0.9156(3)	0.05416(11)	0.87897(17)	0.0441(7)
N1	0.8411(3)	0.19836(13)	0.1271(2)	0.0289(6)
N2	0.6140(3)	0.24351(14)	0.1411(3)	0.0405(7)
N3	0.4853(3)	0.58666(13)	0.2985(3)	0.0370(7)
N4	0.7178(3)	0.62504(12)	0.2795(2)	0.0273(6)
C1	1.0111(4)	0.13934(15)	0.4156(2)	0.0263(7)
C2	0.9401(4)	0.10026(14)	0.5003(2)	0.0232(7)
C3	0.8504(4)	0.04750(15)	0.4694(2)	0.0292(7)
H3A	0.8314	0.0365	0.3956	0.035*
C4	0.7888(4)	0.01093(17)	0.5475(3)	0.0342(8)
H4	0.7268	−0.0241	0.5261	0.041*
C5	0.8196(4)	0.02652(15)	0.6577(2)	0.0288(7)
H5	0.7799	0.0014	0.7103	0.035*
C6	0.9091(4)	0.07927(15)	0.6898(2)	0.0256(7)
C7	0.9679(4)	0.11651(15)	0.6105(2)	0.0261(7)
H7	1.0263	0.1526	0.6315	0.031*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C8	0.9445(4)	0.09539(16)	0.8103(2)	0.0288(7)
C9	0.7505(4)	0.22448(16)	0.1930(3)	0.0346(8)
H9	0.7785	0.2293	0.2682	0.042*
C10	0.6179(5)	0.2289(2)	0.0337(3)	0.0566(12)
H10	0.5393	0.2363	−0.0235	0.068*
C11	0.7574(5)	0.2015(2)	0.0254(3)	0.0505(11)
H11	0.7911	0.1870	−0.0395	0.061*
C12	0.4880(5)	0.27460(19)	0.1907(4)	0.0604(13)
H12A	0.3891	0.2591	0.1548	0.072*
H12B	0.4952	0.2623	0.2675	0.072*
C13	0.4889(7)	0.3440(2)	0.1840(5)	0.097(2)
H13	0.5328	0.3647	0.1281	0.116*
C14	0.4194(5)	0.37989(19)	0.2673(4)	0.0613(12)
H14	0.3826	0.3587	0.3255	0.074*
C15	0.4071(6)	0.4490(2)	0.2599(5)	0.0856(19)
H15	0.4266	0.4692	0.1955	0.103*
C16	0.3662(6)	0.4858(2)	0.3487(4)	0.0724(15)
H16	0.3420	0.4644	0.4109	0.087*
C17	0.3593(4)	0.55532(18)	0.3491(4)	0.0509(11)
H17A	0.3629	0.5698	0.4245	0.061*
H17B	0.2601	0.5686	0.3103	0.061*
C18	0.4813(4)	0.60579(18)	0.1926(3)	0.0451(10)
H18	0.3967	0.6032	0.1382	0.054*
C19	0.6239(4)	0.62928(17)	0.1814(3)	0.0386(8)
H19	0.6539	0.6459	0.1169	0.046*
C20	0.6296(4)	0.59894(16)	0.3478(3)	0.0350(8)
H20	0.6630	0.5901	0.4212	0.042*

(0.2 mmol, 0.0612 g) and distilled water (10 mL) was heated in a 25 mL stainless steel reactor at 423 K for 42 h, followed by slow cooling to room temperature. Green crystals of the title compound formed.

Experimental details

Hydrogen atoms were placed in calculated positions and were included with U_{iso}(H) set to 1.2U_{eq}(C), and 1.5U_{eq}(O).

Discussion

Metalorganic frameworks are of interest in the field of molecular magnetism and materials chemistry [3–9]. The diimidazole ligand with a long spacer are a good choice. The characteristics are as follows: The spacers often causes the structural diversities [10, 11]. Furthermore, the 1,6-di(1*H*-imidazol-1-yl)hexane as a *N*-donor ligand with two potential N coordination sites has rarely been used. Therefore, we selected isophthalic acid as the main ligand [12], and the nitrogen-rich 1,6-di(1*H*-imidazol-1-yl)hexane as a *N*-donor linker, to construct the target compound.

The asymmetric unit of the title structure contains one Ni(II) metal center, one 1,6-di(1*H*-imidazol-1-yl)hexane and one isophthalate monoanion to construct a coordination polymer. The nickel atom is six-coordinated by one oxygen atom from one isophthalate ligand, two nitrogen atoms from two 1,6-di(1*H*-imidazol-1-yl)hexane ligands and three oxygen atoms from three water molecules. The Ni-O bond lengths are in the range of 2.081(2) to 2.108(2) Å, the Ni-N1 bond length is 2.070(3) Å. The bond angles of O—Ni—O are in the range of 88.30(8)° to 177.91(9)°. There are intermolecular hydrogen bonds d(O(1)–H(1W)···O(6)B) = 2.746(3) Å, d(O(2)–H(3W)···O(7)A) = 2.683(3) Å, d(O(3)–H(5W)···O(5)B) = 2.764(3) Å. Symmetry codes: A x, y, $z-1$; B x, $-y+1/2$, $z-1/2$. Thus these interactions result in the infinite three dimensional architecture.

Acknowledgements: This work was supported by the Institutions of higher learning key scientific research project of Henan Province (18B150015) and the Natural Science Foundation of Henan Province (182102310473).

References

1. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
2. Sheldrick, G. M.: Crystal structure refinement with SHELXL. Acta Crystallogr. **C71** (2015) 3–8.
3. Fu, H.-R.; Kang, Y.; Zhang, J.: Highly selective sorption of small hydrocarbons and photocatalytic properties of three metal-organic frameworks based on tris(4-(1*H*imidazol-1-yl)phenyl)amine ligand. Inorg. Chem. **53** (2014) 4209–4214.
4. 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. org. Metal-Org. Nano-Metal Chem. **43** (2013) 710–713.
5. Zheng, B.; Luo, J. H.; Wang, F.; Peng, Y.; Li, G. H.; Huo, Q. S.; Liu, Y. L.: Construction of six coordination polymers based on a 5,50-(1,2-ethynyl)bis-1,3-benzenedicarboxylic ligand: synthesis, structure, gas sorption, and magnetic properties. Cryst. Growth Des. **13** (2013) 1033–1044.
6. 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.
7. Betard, A.; Wannapaiboon, S.; Fischer, R. A.: Assessing the adsorption selectivity of linker functionalized, moisturestable metal-organic framework thin lms by means of an environment-controlled quartz crystal microbalance. Chem. Commun. **48** (2012) 10493–10495.
8. Zhao, H.; Jin, Z.; Su, H.; Zhang, J.; Yao, X.; Zhao, H.; Zhu, G.: Target synthesis of a novel porous aromatic framework and its highly selective separation of CO_2/CH_4. Chem. Commun. **49** (2013) 2780–2782.
9. 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. org. Metal-Org. Nano-Metal Chem. **43** (2013) 1487–1491.

10. Zhang, Z. H.; Chen, S. C.; He, M. Y.; Chen, Q.; Du, M.: Coordination assembly of Zn(II)/Cd(II) terephthalate with bis-pyridinecarboxamide tectons: establishing net entanglements from [3 + 3] interpenetration to high-connected self-penetration. Cryst. Growth Des. **13** (2013) 996–1001.
11. Li, S. H.; Han, M. L.; Liu, G. Z.; Ma, L. F.; Wang, L. Y.: Guest-induced single-crystal-to-single-crystal transformations of a new 4-connected 3D cadmium(II) metal-organic framework. RSC Advances. **5** (2015) 17588–17591.
12. Han, M.-L.; Wang, J.-G.; Ma, L.-F.; Guo, H.; Wang, L.-Y.: Construction of Cd(II) coordination polymers based on R-isophthalate (R = $-CH_3$ or $-OCH_3$) and flexible *N*-donor co-ligands: syntheses, structures and photoluminescence. CrystEngComm **14** (2012) 2691–2701.