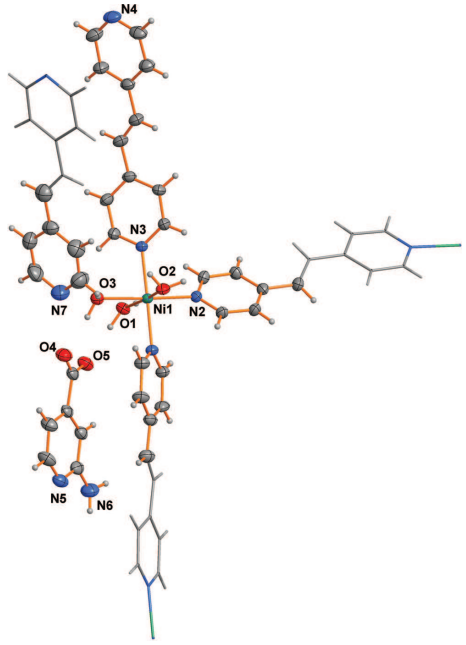
Yu-Fang Wang*, Jing-Jing Wei and Xiao-Hui Zhai

Crystal structure of *catena*-poly[triaqua(μ$_2$-1,2-bis(4-pyridyl)ethane-κ^2*N*:*N*′)-(1,2-bis(4-pyridyl)ethane-κ*N*)nickel(II)] 2-aminonicotinate nitrate – 1,2-bis(4-pyridyl)ethane – water (2/1/8), $C_{36}H_{44}N_8NiO_{12}$



https://doi.org/10.1515/ncrs-2017-0273
Received October 27, 2017; accepted February 14, 2018; available online March 5, 2018

Abstract

$C_{36}H_{44}N_8NiO_{12}$, triclinic, $P\bar{1}$ (no. 2), $a = 10.6451(5)$ Å, $b = 12.1086(6)$ Å, $c = 17.2600(7)$ Å, $\alpha = 76.624(4)°$, $\beta = 72.200(4)°$, $\gamma = 70.802(5)°$, $V = 1979.18(18)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0414$, $wR_{ref}(F^2) = 0.1058$, $T = 293(2)$ K.

CCDC no.: 1824010

A part of the title crystal structure is shown in the figure. The nitrate counteranion as well as the water molecules which are not coordinated to the Ni(II) are omitted for clarity. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Clear green block
Size:	0.20 × 0.18 × 0.18 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.56 mm^{-1}
Diffractometer, scan mode:	SuperNova, EosS2, ω-scans
θ_{max}, completeness:	25.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	22780, 7347, 0.036
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5965
$N(param)_{refined}$:	527
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3]

Source of material

2-Amino-isonicotinic acid (H_3L) (27.6 mg, 0.20 mmol), bis(4-pyridyl)ethane (bpe) (36.4 mg, 0.20 mmol) and $Ni(NO_3)_2{\cdot}6H_2O$ (58.2 mg, 0.20 mmol) were dissolved in H_2O, and stirred. Then the mixture was sealed in a 25 mL Teflon-lined stainless steel autoclave at 130 °C for 72 h, cooled to room temperature at a rate of 5 °C h^{-1}. Green block-shaped crystals were obtained.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Discussion

The metal-organic framework/coordination polymer synthetic investigations have continued unabated over the past decade [4–8]. It drives the design and development of new metal-organic framework materials. In that regard, heterocyclic pyridine-based ligands are perhaps the most promising candidates, because heterocyclic pyridine can interact with a metal surface by both π electrons and the lone-pair electrons at the nitrogen atom, and the incorporation of heterocyclic pyridine ligand provides access to myriad and diverse polymer structural topologies [9–12]. We set about attempts to prepare crystalline divalent transition metal coordination polymers containing the easily prepared 2-aminonicotinate (H_3L–). Additionally, the N-donor ligand 1,2-bi (4-pyridyl)ethylene (bpe) is introduced as secondary ligand to modify the structure of resulting compound.

***Corresponding author: Yu-Fang Wang,** College of Chemistry and Chemical Engineering, and Henan Key Laboratory of Function-Oriented Porous Materials, Luoyang Normal University, Luoyang, Henan 471984, P.R. China, e-mail: wangyf78@163.com
Jing-Jing Wei and Xiao-Hui Zhai: College of Chemistry and Chemical Engineering, and Henan Key Laboratory of Function-Oriented Porous Materials, Luoyang Normal University, Luoyang, Henan 471984, P.R. China

Open Access. © 2018 Yu-Fang Wang et al., published by De Gruyter. BY-NC-ND This work is licensed under the Creative Commons Attribution-NonCommercial-NoDerivatives 4.0 License.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.35491(3)	0.56445(2)	0.24124(2)	0.02566(10)
O1	0.34398(16)	0.69976(13)	0.29868(10)	0.0343(4)
H1A	0.4240	0.7088	0.2883	0.051*
H1B	0.2690	0.7604	0.2993	0.051*
O2	0.33057(17)	0.42851(13)	0.19804(10)	0.0335(4)
H2A	0.3026	0.3782	0.2385	0.050*
H2B	0.4079	0.3927	0.1680	0.050*
O3	0.16360(17)	0.55976(14)	0.33002(10)	0.0376(4)
H3A	0.1310	0.5105	0.3200	0.056*
H3B	0.1067	0.6283	0.3270	0.056*
N1	0.24881(19)	0.68406(16)	0.15766(11)	0.0292(4)
N2	0.54485(18)	0.56253(16)	0.15705(11)	0.0278(4)
N3	0.45748(19)	0.44311(16)	0.32678(11)	0.0286(4)
N4	0.7363(2)	−0.2603(2)	0.66960(15)	0.0493(6)
C1	0.2822(3)	0.7842(2)	0.11999(14)	0.0342(6)
H1	0.3607	0.7955	0.1262	0.041*
C2	0.2048(3)	0.8713(2)	0.07230(15)	0.0387(6)
H2	0.2316	0.9394	0.0470	0.046*
C3	0.0870(3)	0.8568(2)	0.06217(15)	0.0361(6)
C4	0.0561(3)	0.7515(2)	0.09863(16)	0.0405(6)
H4	−0.0198	0.7366	0.0918	0.049*
C5	0.1383(3)	0.6688(2)	0.14508(15)	0.0355(6)
H5	0.1156	0.5985	0.1690	0.043*
C6	−0.0072(3)	0.9481(2)	0.01735(16)	0.0424(6)
H6	−0.0833	0.9298	0.0137	0.051*
C7	0.4247(3)	0.4568(2)	0.40567(14)	0.0349(6)
H7	0.3678	0.5292	0.4209	0.042*
C8	0.4710(3)	0.3691(2)	0.46586(15)	0.0393(6)
H8	0.4446	0.3828	0.5202	0.047*
C9	0.5573(3)	0.2601(2)	0.44483(15)	0.0336(5)
C10	0.5980(3)	0.2490(2)	0.36200(14)	0.0356(6)
H10	0.6601	0.1797	0.3444	0.043*
C11	0.5467(2)	0.3399(2)	0.30607(14)	0.0327(5)
H11	0.5750	0.3297	0.2510	0.039*
C12	0.5948(3)	0.1606(2)	0.50850(15)	0.0411(6)
H12	0.5802	0.1770	0.5611	0.049*
C13	0.6476(3)	0.0499(2)	0.49590(16)	0.0415(6)
H13	0.6669	0.0363	0.4420	0.050*
C14	0.6797(3)	−0.0546(2)	0.55679(16)	0.0391(6)
C15	0.7329(3)	−0.1653(2)	0.53234(17)	0.0443(6)
H15	0.7513	−0.1729	0.4773	0.053*
C16	0.7586(3)	−0.2645(2)	0.59018(19)	0.0474(7)
H16	0.7933	−0.3381	0.5725	0.057*
C17	0.6884(3)	−0.1534(3)	0.69234(18)	0.0550(8)
H17	0.6742	−0.1484	0.7475	0.066*
C18	0.6584(3)	−0.0497(2)	0.63961(16)	0.0477(7)
H18	0.6245	0.0227	0.6591	0.057*
C19	0.5642(2)	0.5583(2)	0.07726(14)	0.0314(5)
H19	0.4892	0.5631	0.0585	0.038*
C20	0.6902(2)	0.5474(2)	0.02198(14)	0.0346(6)
H20	0.6986	0.5458	−0.0330	0.042*
C21	0.8049(2)	0.53862(19)	0.04768(14)	0.0304(5)
C22	0.7837(2)	0.5457(2)	0.13037(14)	0.0335(5)
H22	0.8565	0.5424	0.1506	0.040*
C23	0.6542(2)	0.5574(2)	0.18173(14)	0.0318(5)
H23	0.6421	0.5620	0.2367	0.038*
C24	0.9408(2)	0.5200(2)	−0.01027(15)	0.0346(5)
H24	0.9438	0.5378	−0.0661	0.042*
O4	−0.02962(19)	0.76229(15)	0.32512(11)	0.0461(5)
O5	0.11418(18)	0.87289(15)	0.29722(11)	0.0439(4)
N5	−0.2631(2)	1.14030(19)	0.17073(14)	0.0440(5)
N6	−0.1008(3)	1.2436(2)	0.12465(17)	0.0578(7)
H6A	−0.0114	1.2429	0.1103	0.069*
C25	−0.3019(3)	1.0454(2)	0.21550(19)	0.0493(7)
H25	−0.3889	1.0417	0.2179	0.059*
C26	−0.2222(3)	0.9533(2)	0.25802(18)	0.0447(7)
H26	−0.2546	0.8897	0.2885	0.054*
C27	−0.0918(2)	0.9578(2)	0.25426(14)	0.0324(5)
C28	−0.0491(3)	1.0540(2)	0.20929(15)	0.0356(6)
H28	0.0377	1.0592	0.2060	0.043*
C29	−0.1384(3)	1.1448(2)	0.16813(15)	0.0383(6)
C30	0.0061(3)	0.8566(2)	0.29558(15)	0.0351(6)
N7	−0.1024(3)	0.3932(2)	0.36552(17)	0.0652(7)
C31	−0.1194(3)	0.3760(3)	0.4466(2)	0.0630(9)
H31	−0.1475	0.4421	0.4729	0.076*
C32	−0.0982(3)	0.2669(3)	0.49380(19)	0.0561(8)
H32	−0.1132	0.2608	0.5504	0.067*
C33	−0.0547(3)	0.1663(2)	0.45751(18)	0.0458(7)
C34	−0.0372(3)	0.1836(3)	0.37344(19)	0.0554(8)
H34	−0.0081	0.1190	0.3455	0.066*
C35	−0.0627(4)	0.2960(3)	0.3312(2)	0.0650(9)
H35	−0.0514	0.3047	0.2748	0.078*
C36	−0.0291(3)	0.0508(2)	0.5112(2)	0.0577(8)
H36	−0.0589	0.0527	0.5676	0.069*
O10	0.4009(3)	0.1469(2)	0.0621(2)	0.1053(10)
O11	0.2181(3)	0.1751(2)	0.0247(2)	0.0971(9)
O12	0.2038(3)	0.2388(3)	0.13238(19)	0.1012(9)
N8	0.2728(3)	0.1856(2)	0.0744(2)	0.0678(8)
O6	−0.2002(3)	0.6418(2)	0.3038(2)	0.0920(9)
H6C	−0.1638	0.5683	0.3161	0.138*
H6D	−0.1690	0.6810	0.3247	0.138*
O8	0.5533(3)	0.9090(2)	0.11652(18)	0.1082(10)
H8A	0.4975	0.9758	0.1170	0.162*
H8B	0.5823	0.9003	0.0648	0.162*
O7	0.5520(2)	0.79347(17)	0.27416(14)	0.0624(6)
H7A	0.6298	0.7482	0.2798	0.094*
H7B	0.5524	0.8392	0.2240	0.094*
O9	0.5342(2)	0.29461(16)	0.09332(12)	0.0502(5)
H9A	0.4946	0.2500	0.0842	0.075*
H9B	0.5995	0.2526	0.1152	0.075*
H6B	−0.152(4)	1.288(3)	0.094(2)	0.085(12)*

A perspective view of a part of the title structure $\{[Ni(bpe)_2(H_2O)_3]\cdot 4H_2O\cdot(NO_3)\cdot 0.5(bpe)\cdot(H_2L)\}_n$ is shown in the figure. The complex crystallizes in the triclinic space group $P\bar{1}$ and exhibits a 1D chain structure. The asymmetric unit consists of one Ni(II) ion, two coordinated bpe ligands,

three coordinated water molecules, four uncoordinated water molecules and one uncoordinated nitrate ion, half of uncoordinated bpe, one uncoordinated H_2L^-. The Ni(II) ion in the complex is six-coordinated by three nitrogen atoms [N(1), N(2), N(3)] of three different bpe ligands and three oxygen atoms [O(1), O(2) and O(3)] from three water ligands. The bond lengths of Ni—N and Ni—O are 2.095(2)—2.1116(19) Å and 2.0605(17)—2.1511(18) Å, respectively, which are in the normal range [10]. The O(1)—Ni(1)—N(1) and N(1)—Ni(1)—O(3) bond angles are 90.83(7)° and 90.54(7)°, respectively. In this complex, bpe, acting as terminal ligand, coordinates the Ni(II) ions through a nitrogen atom of the pyrazole rings. Another bpe (two halfs; see the figure) acts as bridging ligand being connected with two Ni(II) ions that are linked to a nitrogen atom of the pyrazole rings [13]. One bpe molecule keeps uncoordinated. The coordination leads to a one-dimensional infinite extending chain. Chains are linked by a system of hydrogen bonds. Classic hydrogen bonds O—H···O and O—H···N link them into a 3D network.

References

1. Agilent Technologies: CrysAlis^{PRO} Software system, version 1.171.38.41r, Agilent Technologies UK Ltd, Oxford, UK (2011).
2. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
3. Sheldrick, G. M.: Crystal structure refinement with SHELXL. Acta Crystallogr. **C71** (2015) 3–8.
4. Chen, Y. Q.; Qu, Y. K.; Li, Z. Z.; Chang, Z.; Hu, T. L.; Xu, J.; Bu, X. H.: Zn(II)-benzotriazolate clusters based amide functionalized porous coordination polymers with high CO_2 adsorption selectivity. Inorg. Chem. **53** (2014) 8842–8844.
5. He, Y. B.; Zhou, W.; Qi, G. D.; Chen, B. L.: Methane storage in metal–organic frameworks. Chem. Soc. Rev. **43** (2014) 5657–5678.
6. Wang, G. Y.; Yang, L. L.; Song, H.; Ruan, W. J.; Chang, Z.; Bu, X. H.: A luminescent 2D coordination polymer for selective sensing of nitrobenzene. Dalton Trans. **42** (2013) 12865–12868.
7. Zhang, X. Q.; Chi, Z. G.; Zhang, Y.; Liu, S. W.; Xu, J. R.: Recent advances in mechanochromic luminescent metal complexes. J. Mater. Chem. **1** (2013) 3376–3390.
8. Erer, H.; Yesile, O. Z.; Arici, M.; Keskin, S.; Buyukgungorc, O.: 2D-3D polycatenated and 3D-3D interpenetrated metal-organic frameworks constructed from thiophene-2,5-dicarboxylate and rigid bis(imidazole) ligands. O. J. Solid State Chem. **210** (2014) 261–266.
9. Timokhin, I.; Pettinari, C.; Marchetti, F.; Pettinari, R.; Condello, F.; Galli, S.; Alegria, E. C. B. A.; Martins, L. M. D. R. S.; Pombeiro, A. J. L.: Novel coordination polymers with (pyrazolato)-based tectons: catalytic activity in the peroxidative oxidation of alcohols and cyclohexane. Cryst. Growth Des. **15** (2015) 2303–2317.
10. Walter, S. M.; Kniep, F.; Herdtweck, E.; Huber, S. M.: Halogen-bond-induced activation of a carbon–heteroatom Bond. Chem. Int. Ed. **50** (2011) 7187–7191.
11. Wang, K.; Chen, Z. L.; Zou, H. H.; Zhang, Z.; Sun, W. Y.; Liang, F. P.: Two types of Cu-Ln heterometallic coordination polymers with 2-hydroxyisophthalate: syntheses, structures, and magnetic properties. Cryst. Growth Des. **15** (2015) 2883–2890.
12. Gao, J. G.; Ye, K. Q.; He, M.; Xiong, W. W.; Cao, W. F.; Le, Z. Y.; Wang, Y.; Wu, T.; Huo, F. W.; Liu, X. G.; Zhang, Q. C.: Tuning metal–carboxylate coordination in crystalline metal–organic frameworks through surfactant media. J. Solid State Chem. **206** (2013) 27–31.
13. Yin, W.-D.; Li, G. L.: Crystal structure of poly[aqua(1,2-bis(4-pyridyl)ethane-κ^2N:N')(4-nitro-1,2-benzenedicarboxylate-κ^3O,O':O'')cobalt(II)], $C_{20}H_{17}CoN_3O_7$. Z. Kristallogr. NCS **230** (2015) 11–12.