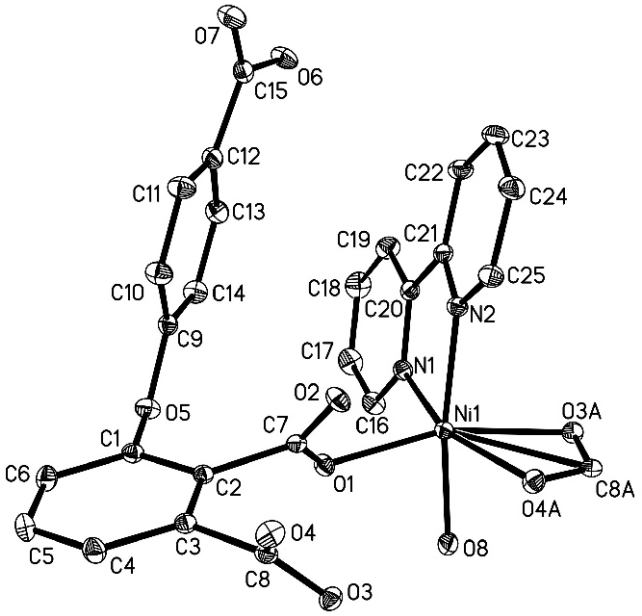
Crystal structure of *catena*-aqua(2,2′-bipyridine)(3-(4-carboxy-phenoxy)phthalato)nickel(II), $Ni(H_2O)(C_{10}H_8N_2)(C_{15}H_8O_7)$

Xiao-Dong Li[*,I,II], Kun Wang[I] and Feng-Feng Guo[I]

[I] Department of Chemistry, Zhengzhou University, Zhengzhou 450052, Henan, P. R. China
[II] Luoyang Normal University, College of Chemistry and Chemical Engineering, Luoyang 471022, Henan, P. R. China

Received May 20, 2011, accepted and available on-line September 14, 2011; CCDC no. 1267/3582



Abstract

$C_{25}H_{18}N_2NiO_8$, orthorhombic, *Pbca* (no. 61), a = 9.9298(7) Å, b = 18.029(1) Å, c = 24.512(2) Å, V = 4388.4 Å^3, Z = 8, $R_{gt}(F)$ = 0.026, $wR_{ref}(F^2)$ = 0.069, T = 296 K.

Source of material

The title compound was synthesized by the reaction of $Ni(OAc)_2 \cdot 4H_2O$ (0.01 mmol), 3-(4-carboxy-phenoxy)phthalic acid (H_3L) (0.01 mmol), 2,2′-bipyridine (2,2′-bipy, 0.01 mmol), and NaOH (0.0020 g) in the molar ratio of 1:1:1 in 15 ml water. The reaction was carried out in a sealed Teflon-lined stainless steel autoclave reactor at 140 °C for 3 days. After the mixture was cooled to room temperature, blue crystals were collected.

Discussion

The design and synthesis of metal-organic frameworks (MOFs) have attracted considerable attention because of their potential applications as function materials as well as their structural diversity and intriguing variety of topologies [1-6]. In principle, the most effective approach for the construction of MOFs is to rationally modify the building blocks and to control the assembled motifs for required products via selecting different organic ligands. It is well known that carboxylate ligands play an important role in coordination chemistry, which may adopt diverse binding modes such as monodentate, chelating, and bridging in the *syn-syn*, *syn-anti*, and *anti-anti* configurations. 3-(4-carboxy-phenoxy)phthalic acid (H_3L), as an *O*-donor ligand with three coordinating carboxylic groups attached at asymmetrical positions of a semirigid V-shaped central molecular framework, has been used to construct MOFs [7].

The asymmetric unit of the title crystal structure contains one Ni(II) ion, one HL ligand, one 2,2′-bipy ligand, and one coordinated water molecule. The Ni(II) ion is six-coordinated by two nitrogen atoms of one 2,2′-bipy ligand, three oxygen atoms of two HL ligands, and one oxygen atom from coordinated water molecule, resulting in a distorted octahedral coordination. The Ni—O and Ni—N bond lengths are in the range of 2.054(1) - 2.194(1) Å and 2.058(2) - 2.062(2) Å, respectively, which are in the normal range of other related Ni(II) complexes. The partly deprotonated HL ligand acts in monodentate and bidentate-chelating coordination modes to bridge the adjacent Ni(II) ions yielding a zigzag chain, and the dihedral angle of two aromatic rings is 77.35°. The Ni···Ni separation across the HL ligand is 5.8337(3) Å. It is worth noting that the individual zigzag chains are connected into a 3D intricate network via hydrogen bonding interactions. Despite the presence of phenyl moieties in 2,2′-bipy and HL ligands, the title crystal structure does not exhibit $\pi\cdots\pi$ stacking interactions.

Table 1. Data collection and handling.

Crystal:	blue block, size 0.18 × 0.21 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.42 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	31544, 4069
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3385
$N(param)_{refined}$:	326
Programs:	SHELXS-97, SHELXL-97, SHELXTL [8]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(6)	8*c*	0.7414	–0.1993	0.3640	0.059
H(1W)	8*c*	0.5510	0.2856	0.4242	0.055
H(2W)	8*c*	0.5277	0.2942	0.4853	0.055
H(4)	8*c*	1.2864	0.3245	0.3466	0.038
H(5)	8*c*	1.2428	0.3001	0.2559	0.045
H(6A)	8*c*	1.0579	0.2296	0.2315	0.041
H(10)	8*c*	1.0737	0.0857	0.3239	0.042
H(11)	8*c*	1.0613	–0.0398	0.3421	0.044
H(13)	8*c*	0.6696	–0.0396	0.3038	0.040
H(14)	8*c*	0.6818	0.0856	0.2844	0.040
H(16)	8*c*	0.4459	0.1989	0.3766	0.044
H(17)	8*c*	0.3175	0.1253	0.3206	0.053

* Correspondence author (e-mail: dongxiaoli@lynu.edu.cn)

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(18)	8*c*	0.3319	–0.0025	0.3290	0.055
H(19)	8*c*	0.4804	–0.0531	0.3918	0.046
H(22)	8*c*	0.6211	–0.0921	0.4544	0.043
H(23)	8*c*	0.7828	–0.1248	0.5182	0.046
H(24)	8*c*	0.9141	–0.0332	0.5573	0.044
H(25)	8*c*	0.8781	0.0888	0.5335	0.037

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni(1)	8*c*	0.66704(2)	0.17847(1)	0.466304(9)	0.0223(1)	0.0203(1)	0.0237(1)	–0.00083(9)	0.0003(1)	0.0010(1)
O(1)	8*c*	0.7921(1)	0.21547(7)	0.40447(5)	0.0214(7)	0.0291(7)	0.0290(7)	0.0015(6)	0.0024(6)	0.0060(6)
O(2)	8*c*	0.9721(1)	0.16479(7)	0.44432(6)	0.0284(8)	0.0332(8)	0.0326(7)	0.0009(6)	–0.0022(6)	0.0127(6)
O(3)	8*c*	1.0543(1)	0.32617(7)	0.46055(5)	0.0278(7)	0.0284(7)	0.0272(7)	0.0055(6)	0.0008(6)	–0.0019(6)
O(4)	8*c*	1.2570(1)	0.27776(7)	0.45854(5)	0.0255(7)	0.0334(8)	0.0344(8)	0.0036(6)	–0.0066(6)	–0.0035(6)
O(5)	8*c*	0.8767(1)	0.17271(7)	0.29191(5)	0.0359(8)	0.0258(7)	0.0304(7)	–0.0017(6)	–0.0099(6)	0.0016(6)
O(6)	8*c*	0.7372(2)	–0.15805(8)	0.35000(6)	0.0372(9)	0.0310(8)	0.0504(9)	–0.0061(7)	–0.0004(7)	0.0124(7)
O(7)	8*c*	0.9609(2)	–0.16599(8)	0.35513(7)	0.0362(9)	0.0354(8)	0.058(1)	0.0013(7)	0.0025(8)	0.0150(7)
O(8)	8*c*	0.5702(2)	0.27842(7)	0.45753(6)	0.0462(9)	0.0333(8)	0.0303(8)	0.0105(7)	0.0091(7)	0.0032(6)
N(1)	8*c*	0.5422(2)	0.11952(9)	0.41493(6)	0.0250(8)	0.0281(9)	0.0270(9)	–0.0022(7)	0.0001(7)	0.0016(7)
N(2)	8*c*	0.7307(2)	0.07188(8)	0.48167(6)	0.0255(9)	0.0217(8)	0.0286(8)	–0.0005(7)	0.0016(7)	0.0016(7)
C(1)	8*c*	0.9882(2)	0.2149(1)	0.30817(8)	0.028(1)	0.0223(9)	0.026(1)	0.0015(8)	–0.0028(8)	0.0009(8)
C(2)	8*c*	1.0087(2)	0.23093(9)	0.36308(7)	0.0218(9)	0.0195(9)	0.0240(9)	0.0040(7)	0.0007(8)	0.0025(7)
C(3)	8*c*	1.1228(2)	0.2725(1)	0.37691(7)	0.024(1)	0.0211(9)	0.026(1)	0.0037(7)	0.0000(8)	0.0013(7)
C(4)	8*c*	1.2104(2)	0.2974(1)	0.33688(8)	0.027(1)	0.032(1)	0.035(1)	–0.0041(8)	0.0048(9)	0.0022(9)
C(5)	8*c*	1.1850(2)	0.2822(1)	0.28268(9)	0.041(1)	0.040(1)	0.030(1)	–0.003(1)	0.013(1)	0.0049(9)
C(6)	8*c*	1.0743(2)	0.2405(1)	0.26797(8)	0.042(1)	0.038(1)	0.022(1)	0.001(1)	0.0025(9)	–0.0007(8)
C(7)	8*c*	0.9185(2)	0.2008(1)	0.40788(7)	0.025(1)	0.0178(9)	0.025(1)	–0.0007(7)	0.0009(8)	–0.0006(7)
C(8)	8*c*	1.1473(2)	0.2930(1)	0.43548(8)	0.026(1)	0.0185(9)	0.028(1)	–0.0023(8)	0.0011(8)	0.0009(8)
C(9)	8*c*	0.8782(2)	0.0979(1)	0.30362(7)	0.032(1)	0.027(1)	0.0217(9)	–0.0011(8)	–0.0002(8)	0.0000(8)
C(10)	8*c*	0.9923(2)	0.0606(1)	0.32021(9)	0.026(1)	0.031(1)	0.048(1)	–0.0049(9)	–0.0026(9)	0.0027(9)
C(11)	8*c*	0.9843(2)	–0.0144(1)	0.33125(9)	0.028(1)	0.031(1)	0.051(1)	0.0022(9)	–0.004(1)	0.004(1)
C(12)	8*c*	0.8638(2)	–0.0525(1)	0.32639(8)	0.031(1)	0.027(1)	0.028(1)	–0.0025(8)	0.0015(8)	0.0010(8)
C(13)	8*c*	0.7506(2)	–0.0145(1)	0.30824(8)	0.027(1)	0.035(1)	0.037(1)	–0.0066(9)	–0.0009(9)	0.0010(9)
C(14)	8*c*	0.7577(2)	0.0605(1)	0.29672(8)	0.028(1)	0.035(1)	0.036(1)	0.0018(9)	–0.0057(9)	0.0015(9)
C(15)	8*c*	0.8593(2)	–0.1309(1)	0.34466(8)	0.033(1)	0.031(1)	0.026(1)	–0.0048(9)	0.0012(9)	0.0006(8)
C(16)	8*c*	0.4543(2)	0.1476(1)	0.37911(9)	0.035(1)	0.038(1)	0.037(1)	–0.003(1)	–0.006(1)	0.0044(9)
C(17)	8*c*	0.3757(2)	0.1039(1)	0.34583(9)	0.039(1)	0.055(2)	0.038(1)	–0.006(1)	–0.011(1)	0.004(1)
C(18)	8*c*	0.3849(2)	0.0282(1)	0.35062(9)	0.040(1)	0.053(2)	0.044(1)	–0.015(1)	–0.010(1)	–0.010(1)
C(19)	8*c*	0.4736(2)	–0.0019(1)	0.38781(9)	0.037(1)	0.034(1)	0.045(1)	–0.008(1)	–0.000(1)	–0.007(1)
C(20)	8*c*	0.5528(2)	0.0452(1)	0.41918(8)	0.025(1)	0.027(1)	0.029(1)	–0.0039(8)	0.0051(8)	–0.0025(8)
C(21)	8*c*	0.6553(2)	0.0185(1)	0.45839(8)	0.025(1)	0.0230(9)	0.030(1)	–0.0003(8)	0.0067(8)	–0.0006(8)
C(22)	8*c*	0.6735(2)	–0.0559(1)	0.47107(9)	0.036(1)	0.024(1)	0.048(1)	–0.0018(9)	0.006(1)	–0.0011(9)
C(23)	8*c*	0.7702(2)	–0.0753(1)	0.50870(9)	0.041(1)	0.025(1)	0.050(1)	0.006(1)	0.008(1)	0.007(1)
C(24)	8*c*	0.8476(2)	–0.0211(1)	0.53208(9)	0.035(1)	0.036(1)	0.039(1)	0.0087(9)	–0.001(1)	0.009(1)
C(25)	8*c*	0.8253(2)	0.0520(1)	0.51759(8)	0.029(1)	0.030(1)	0.035(1)	0.0014(9)	–0.0027(9)	0.0021(9)

References

1. Guo, X. D.; Zhu, G. S.; Fang, Q. R.; Xue, M.; Tian, G.; Sun, J. Y.; Li, X. T.; Qiu, S. L.: Synthesis, Structure and Luminescent Properties of Rare Earth Coordination Polymers Constructed from Paddle-Wheel Building Blocks. Inorg. Chem. **44** (2005) 3850-3855.
2. Zhao, B.; Cheng, P.; Chen, X. Y.; Shi, W.; Liao, D. Z.; Yan, S. P.; Jiang, Z. H.: Design and Synthesis of 3*d*-4*f* Metal-Based Zeolite-type Materials with a 3D Nanotubular Structure Encapsulated "Water" Pipe. J. Am. Chem. Soc. **126** (2004) 3012-3013.
3. Li, D. S.; Wang, Y. Y.; Luan, X. J; Liu, P.; Zhou, C. H.; Ma, H. R.; Shi, Q. H.: 1-D Open-Channeled 3-D Supramolecular Self-Assembled Frameworks Encapsulating Unprecedented Cyclic $(H_2O)_8$ Clusters or Solvent Molecules. Eur. J. Inorg. Chem. (2005) 2678-2684.
4. Ma, L. F.; Wang, L. Y.; Huo, X. K.; Wang, Y. Y.; Fan, Y. T.; Wang, J. G.; Chen, S. H.: Chain, Pillar, Layer, and Different Pores: A *N*-[(3-Carboxyphenyl)-sulfonyl]glycine Ligand as a Versatile Building Block for the Construction of Coordination Polymers. Cryst. Growth Des. **8** (2008) 620-628.
5. Halper, S. R.; Cohen, S. M.: Self-Assembly of Two Distinct Supramolecular Motifs in a Single Crystalline Framework. Angew. Chem. Int. Ed. **43** (2004) 2385-2388.
6. Ghosh, S. K.; Bharadwaj, P. K.: Puckered-Boat ConformationHexameric Water Clusters Stabilized in a 2D Metal-Organic Framework Structure Built from Cu(II) and 1,2,4,5-Benzenetetracarboxylic Acid. Inorg. Chem. **43** (2004) 5180-5182.
7. Wang, H. L.; Zhang, D. P.; Sun, D. F.; Chen, Y. T.; Zhang, L. F.; Tian, L. J.; Jiang, J. Z.; Ni, Z. H.: Co(II) Metal-Organic Frameworks (MOFs) Assembled from Asymmetric Semirigid Multicarboxylate Ligands: Synthesis, Crystal Structures, and Magnetic Properties. Cryst. Growth Des. **9** (2009) 5273-5282.
8. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.