Crystal structure of diaqua(bis(3,5-bis(isonicotinamido))-benzoato)nickle(II) tetrahydrate, $Ni(H_2O)_2(C_{19}H_{13}N_4O_4)_2 \cdot 4H_2O$

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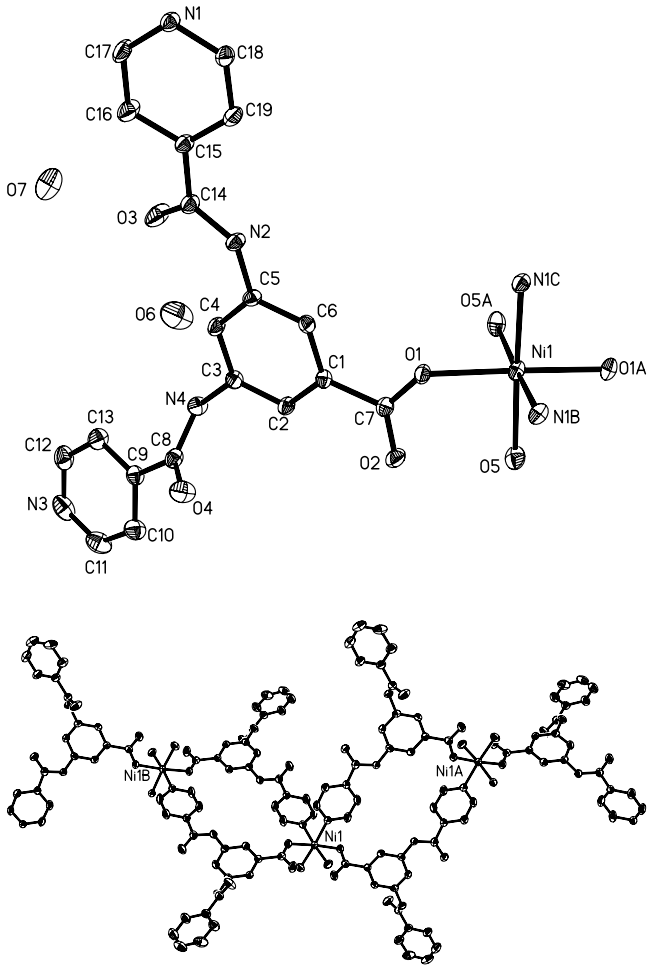

Abstract

$C_{38}H_{38}N_8NiO_{14}$, monoclinic, *C*12/*c*1 (no. 15), a = 17.466(2) Å, b = 10.706(1) Å, c = 21.681(2) Å, β = 104.837(1)°, V = 3919.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.039, $wR_{ref}(F^2)$ = 0.063, T = 293 K.

Source of material

0.1 mmol 3,5-bis(isonicotinamido)benzoic acid (HL) and 0.05 mmol $Ni(NO_3)_2$ were dissolved in 10 ml distilled water. The pH value was adjusted to about 6 with 1 M NaOH solution. Then the mixture was placed in a Teflon-lined stainless vessel (25 ml). The vessel was sealed and heated at 413 K for 72 h under autogenous pressure and then cooled down to room temperature. The resulting solution was filtered and green block-shaped single crystals suitable for analysis were obtained.

Experimental details

Hydrogen atoms bound to carbon atoms were placed in calculated positions and treated as riding on their parent atoms, with d(C—H) = 0.93Å with U_{iso}(H) = 1.2 U_{eq}(C). Hydrogen atoms attached to oxygen of water molecule were initially located in a difference Fourier map and refined.

Discussion

In recent years, the design and synthesis of functional coordination polymers are of great interest due to their intriguing structural topologies and versatile chemical properties [1-4].

The Ni1 atom in the title complex sits on the inversion center and is coordinated by two N atoms from two different bis(3,5-bis(isonicotinamido))benzoate ligands (L^-) and two O atoms from two water molecules, forming a distorted octahedron (figure, top). The coordination bond lengths and angles around the Ni^{II} are in the range of 2.057(1) - 2.105(2) Å and 86.62(6)° - 175.50(9)°, respectively. More interestingly, two adjacent Ni ions linked by the L^- ligand, which the L^- ligand in turn uses its carboxylate group and one of the two pyridinyl groups to connect two metal centers, and another pyridinyl group is free of coordination (figure, bottom). There are macrocyclic rings consisted of 24-membered ring in the infinite chain, which similar to the complex $[Mn(BBA)_2(H_2O)_2] \cdot 4H_2O$ [5]. It is worthwhile to note that hydrogen bonding interactions are usually important in the synthesis of supramolecular architecture. There exist N–H···O and O–H···O hydrogen bonding interactions between crystal molecules and carboxylate oxygen atoms. These hydrogen bonds in the crystal structure, which further link the 1D complex into a three-dimensional framework structure.

Table 1. Data collection and handling.

Crystal:	green block, size 0.10 × 0.14 × 0.18 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	5.76 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10559, 3850
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2359
$N(param)_{refined}$:	296
Programs:	SHELXS-97 [6], SHELXL-97 [7]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(12)	8*f*	0.4343	0.1402	0.7051	0.059
H(11)	8*f*	0.5146	0.0605	0.5592	0.065
H(10)	8*f*	0.4459	0.2226	0.5015	0.053
H(13)	8*f*	0.3604	0.3013	0.6514	0.051
H(2)	8*f*	0.2256	0.4775	0.4217	0.033
H(6)	8*f*	0.1123	0.7905	0.4475	0.032
H(4)	8*f*	0.2219	0.5832	0.5985	0.036
H(19)	8*f*	0.0459	0.9758	0.5770	0.047
H(18)	8*f*	0.0010	1.1421	0.6230	0.047
H(16)	8*f*	0.1739	0.8737	0.7490	0.054
H(17)	8*f*	0.1247	1.0432	0.7906	0.053
H(5B)	8*f*	0.033(2)	0.556(2)	0.186(1)	0.07(1)
H(2A)	8*f*	0.1242	0.8644	0.5488	0.036
H(4A)	8*f*	0.2583	0.3578	0.5629	0.040
H(5A)	8*f*	0.0975	0.5904	0.2341	0.058
H(6B)	8*f*	0.214(2)	0.246(3)	0.650(2)	0.13(2)
H(7B)	8*f*	0.211(2)	0.562(4)	0.800(2)	0.18(2)
H(7A)	8*f*	0.215(2)	0.648(4)	0.758(2)	0.13(2)
H(6A)	8*f*	0.150(2)	0.333(3)	0.634(2)	0.11(2)

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}
C(12)	8*f*	0.4365(2)	0.1564(3)	0.6634(1)	0.049(2)	0.053(2)	0.042(2)	0.001(2)	0.002(2)	0.016(2)
C(11)	8*f*	0.4836(2)	0.1106(2)	0.5783(2)	0.048(2)	0.039(2)	0.077(3)	0.013(2)	0.021(2)	0.007(2)
C(10)	8*f*	0.4419(2)	0.2073(2)	0.5428(1)	0.045(2)	0.041(2)	0.047(2)	0.012(1)	0.014(2)	0.010(1)
C(9)	8*f*	0.3941(1)	0.2808(2)	0.5700(1)	0.033(2)	0.028(2)	0.035(2)	0.004(1)	0.005(1)	0.003(1)
C(13)	8*f*	0.3918(1)	0.2540(2)	0.6314(1)	0.051(2)	0.039(2)	0.037(2)	0.013(2)	0.009(1)	0.011(2)
C(8)	8*f*	0.3507(2)	0.3897(2)	0.5339(1)	0.042(2)	0.030(2)	0.027(2)	0.007(1)	0.006(1)	0.000(1)
C(3)	8*f*	0.2307(1)	0.5151(2)	0.5136(1)	0.033(2)	0.023(1)	0.029(2)	0.007(1)	0.006(1)	0.002(1)
C(2)	8*f*	0.2098(1)	0.5340(2)	0.4486(1)	0.035(2)	0.022(1)	0.023(2)	0.004(1)	0.005(1)	–0.002(1)
C(1)	8*f*	0.1652(1)	0.6375(2)	0.4235(1)	0.027(1)	0.023(1)	0.023(1)	–0.001(1)	0.003(1)	–0.000(1)
C(7)	8*f*	0.1392(1)	0.6577(2)	0.3520(1)	0.031(2)	0.025(1)	0.026(2)	–0.002(1)	0.005(1)	–0.002(1)
C(6)	8*f*	0.1424(1)	0.7209(2)	0.4642(1)	0.027(1)	0.027(2)	0.026(1)	0.006(1)	0.003(1)	0.002(1)
C(5)	8*f*	0.1640(1)	0.7022(2)	0.5297(1)	0.032(2)	0.023(1)	0.026(2)	0.002(1)	0.010(1)	–0.003(1)
C(4)	8*f*	0.2079(1)	0.5976(2)	0.5547(1)	0.039(2)	0.031(1)	0.020(1)	0.006(1)	0.007(1)	0.004(1)
C(14)	8*f*	0.1461(1)	0.7912(2)	0.6310(1)	0.038(2)	0.032(2)	0.024(2)	0.002(1)	0.009(1)	0.002(1)
C(15)	8*f*	0.1150(1)	0.9049(2)	0.6572(1)	0.036(2)	0.028(1)	0.022(1)	0.003(1)	0.009(1)	0.001(1)
C(19)	8*f*	0.0630(2)	0.9871(2)	0.6209(1)	0.050(2)	0.048(2)	0.020(2)	0.015(1)	0.007(1)	–0.003(1)
C(18)	8*f*	0.0358(2)	1.0866(2)	0.6490(1)	0.046(2)	0.049(2)	0.021(2)	0.021(1)	0.005(1)	0.005(1)
C(16)	8*f*	0.1383(2)	0.9270(2)	0.7222(1)	0.069(2)	0.040(2)	0.025(2)	0.024(2)	0.008(2)	0.005(1)
C(17)	8*f*	0.1081(2)	1.0289(2)	0.7469(1)	0.064(2)	0.046(2)	0.018(2)	0.018(2)	0.001(1)	–0.002(1)
N(1)	8*f*	0.0563(1)	1.1078(2)	0.71133(9)	0.038(1)	0.025(1)	0.022(1)	0.004(1)	0.008(1)	0.0012(9)
N(2)	8*f*	0.1421(1)	0.7961(2)	0.56801(9)	0.043(1)	0.026(1)	0.023(1)	0.0109(9)	0.010(1)	0.0019(9)
N(4)	8*f*	0.2777(1)	0.4103(2)	0.54093(9)	0.042(1)	0.026(1)	0.033(1)	0.008(1)	0.013(1)	0.008(1)
N(3)	8*f*	0.4822(1)	0.0847(2)	0.6383(1)	0.041(2)	0.042(2)	0.068(2)	0.004(1)	0.005(1)	0.022(1)
Ni(1)	4*e*	0	0.75084(4)	¼	0.0353(3)	0.0237(2)	0.0191(2)	0	0.0042(2)	0
O(7)	8*f*	0.2326(2)	0.6370(2)	0.7928(1)	0.094(2)	0.054(2)	0.049(2)	–0.019(1)	0.015(2)	0.002(1)
O(6)	8*f*	0.1891(2)	0.3113(3)	0.6265(1)	0.092(2)	0.106(2)	0.097(2)	0.030(2)	0.046(2)	0.057(2)
O(3)	8*f*	0.1707(1)	0.7005(2)	0.66441(8)	0.090(2)	0.035(1)	0.028(1)	0.023(1)	0.018(1)	0.0074(9)
O(4)	8*f*	0.3831(1)	0.4558(2)	0.50181(8)	0.055(1)	0.046(1)	0.053(1)	0.015(1)	0.025(1)	0.024(1)
O(2)	8*f*	0.1670(1)	0.5876(2)	0.31713(7)	0.051(1)	0.046(1)	0.026(1)	0.0172(9)	0.0099(9)	–0.0051(9)
O(1)	8*f*	0.08919(8)	0.7433(1)	0.33292(7)	0.036(1)	0.0304(9)	0.0201(9)	0.0084(9)	0.0018(7)	–0.0005(8)
O(5)	8*f*	0.0607(1)	0.6211(2)	0.20694(8)	0.046(1)	0.031(1)	0.034(1)	0.0012(9)	0.001(1)	–0.0092(9)

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