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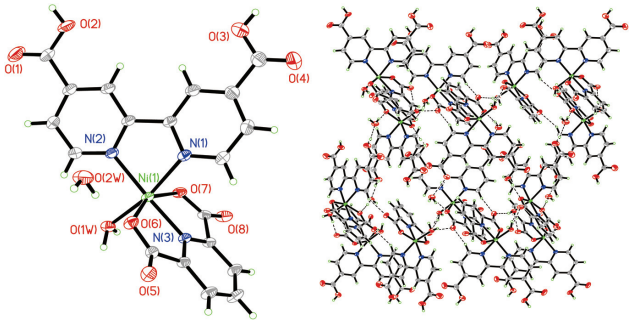
Crystal structure of biaqua(2,2′-bipyridine-4,4′-dicarboxylato-κ²*N*,*N*′)(pyridine-2,6-dicarboxylato-κ³*O*,*N*,*O*′)nickel(II) hydrate, $C_{19}H_{15}N_3NiO_{10}$



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Abstract

$C_{19}H_{15}N_3NiO_{10}$, monoclinic, $P2_1/c$ (no. 14), $a = 12.7175(10)$ Å, $b = 10.6780(10)$ Å, $c = 15.0802(16)$ Å, $\beta = 96.786(9)°$, $V = 2033.5(3)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0358$, $wR_{ref}(F^2) = 0.0960$, $T = 293(2)$ K.

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The molecular 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

The $Ni(CH_3COO)_2$ (0.08840 g, 0.5 mmol) and pyridine-2,6-dicarboxylic acid (0.04187 g, 0.25 mmol) in 10 mL H_2O/methanol (1:4, *v*/*v*) reacted for one and a half hours. And 2,2′-bipyridine-4,4′-dicarboxylic acid (0.2442 g, 1 mmol) was put into the solution. The filtrate was kept at 293 K to obtain green block crystals.

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Table 1: Data collection and handling.

Crystal:	Green block
Size:	0.24 × 0.22 × 0.20 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	1.02 mm^{-1}
Diffractometer, scan mode:	XtaLAB Pro, ω
θ_{max}, completeness:	29.9°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	22810, 5266, 0.029
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4244
$N(param)_{refined}$:	302
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3], WinGX/ORTEP [4]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.84135(2)	0.36710(2)	0.69566(2)	0.02922(8)
C1	0.70667(17)	0.4616(2)	0.83749(16)	0.0438(5)
H1	0.769195	0.461094	0.876300	0.053*
C2	0.61472(18)	0.5023(3)	0.86990(17)	0.0467(5)
H2	0.615442	0.528650	0.928750	0.056*
C3	0.52209(17)	0.5024(2)	0.81175(16)	0.0422(5)
C4	0.52411(16)	0.4613(2)	0.72554(16)	0.0419(5)
H4	0.462215	0.459850	0.685974	0.050*
C5	0.61879(15)	0.4222(2)	0.69800(15)	0.0353(4)
C6	0.62660(15)	0.3810(2)	0.60602(15)	0.0353(4)
C7	0.53850(17)	0.3548(2)	0.54547(16)	0.0424(5)
H5	0.470943	0.359442	0.563060	0.051*
C8	0.55168(19)	0.3218(2)	0.45917(16)	0.0441(5)
C9	0.6533(2)	0.3186(2)	0.43498(16)	0.0466(5)
H7	0.664433	0.301315	0.376353	0.056*
C10	0.73795(18)	0.3413(2)	0.49905(15)	0.0413(5)
H6	0.806100	0.335617	0.482823	0.050*
C11	0.41925(18)	0.5453(2)	0.84244(17)	0.0465(5)
C12	0.4585(2)	0.2899(2)	0.39212(18)	0.0520(6)
C13	1.03089(14)	0.39946(18)	0.61165(13)	0.0305(4)
C14	1.11847(17)	0.3659(2)	0.57033(17)	0.0422(5)
H9	1.166808	0.425989	0.556378	0.051*
C15	1.13241(19)	0.2408(2)	0.55026(18)	0.0493(6)
H10	1.189710	0.216489	0.521283	0.059*
C16	1.06112(17)	0.1522(2)	0.57333(17)	0.0409(5)
H11	1.069421	0.068036	0.559849	0.049*
C17	0.97737(14)	0.19152(18)	0.61682(14)	0.0309(4)
C18	1.00001(15)	0.53119(18)	0.63515(13)	0.0310(4)
C19	0.89373(14)	0.10798(18)	0.64980(15)	0.0324(4)

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
N1	0.72618(13)	0.37106(16)	0.58367(12)	0.0330(3)
N2	0.71014(12)	0.42301(17)	0.75361(12)	0.0347(4)
N3	0.96324(12)	0.31244(15)	0.63390(11)	0.0284(3)
O1	0.41016(17)	0.5689(3)	0.91797(14)	0.0781(7)
O2	0.34376(14)	0.5535(2)	0.77592(13)	0.0650(6)
H3	0.291102	0.584812	0.793466	0.097*
O3	0.36887(16)	0.2950(3)	0.42567(15)	0.0709(6)
H8	0.320167	0.280056	0.386456	0.106*
O4	0.46925(18)	0.2639(2)	0.31612(14)	0.0711(6)
O5	1.05591(13)	0.61827(14)	0.61420(11)	0.0425(4)
O6	0.91722(11)	0.54046(13)	0.67501(10)	0.0362(3)
O7	0.82414(10)	0.16576(13)	0.68847(10)	0.0357(3)
O8	0.89770(12)	−0.00642(14)	0.63829(13)	0.0482(4)
O1W	0.92419(11)	0.35060(13)	0.82186(10)	0.0365(3)
H1A	0.940876	0.272703	0.833419	0.055*
H1B	0.985521	0.387594	0.824409	0.055*
O2W	0.79751(14)	0.75986(18)	0.68385(14)	0.0595(5)
H2A	0.842230	0.700438	0.686287	0.089*
H2B	0.828800	0.828988	0.677110	0.089*

Experimental details

Data of crystal were collected *via* CrysAlis^{PRO} at 293 K [1], the structure was solved by SHELXT [2] program and refined by SHELXL [3] within WinGX [4].

Comment

As is shown in the left part of picture, the Ni1 is six-coordinated by one oxygen atom from water, two nitrogen atoms from one 2,2′-bipyridine-4,4′-dicarboxylic acid ligand, one nitrogen atom and two oxygen atoms from the pyridine-2,6-dicarboxylato ligand (*cf*. the figure). Thus, there is one free water molecule. Most importantly, the weak interactions such as CH···O, OH···O could be found and make the crystal structure become 3D supermolecule structure. All geometric parameters are in the expected ranges and agree with an analogous Cu complex [5].

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