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Crystal structure of tetraaqua-bis(3,5-di(pyridin-4-yl)-1,2,4-triazol-1-ido- κ^1N)nickel(II) dihydrate, $C_{24}H_{28}O_6N_{10}Ni$

Table 1: Data collection and handling.

Crystal:	Green block
Size:	0.45 × 0.39 × 0.30 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.81 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
θ_{max} , completeness:	25.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	9097, 2395, 0.028
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 2105
$N(param)_{refined}$:	203
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3]

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

Atom	x	y	z	U_{iso}^*/U_{eq}
Ni1	1.0000	0.0000	0.5000	0.01874(16)
N1	0.9525(3)	0.13050(13)	0.51811(19)	0.0213(5)
N2	0.7633(3)	0.45049(13)	0.46359(19)	0.0204(5)
N3	0.8267(4)	0.51511(14)	0.6407(2)	0.0276(5)
N4	0.8952(3)	0.43374(14)	0.6595(2)	0.0260(5)
N5	0.5284(4)	0.76152(15)	0.3658(2)	0.0336(6)
C1	0.8862(4)	0.18054(17)	0.4233(2)	0.0262(6)
H1	0.8588	0.1554	0.3487	0.031*
C2	0.8560(4)	0.26713(17)	0.4294(2)	0.0266(6)
H2	0.8100	0.2989	0.3603	0.032*
C3	0.8946(4)	0.30677(15)	0.5391(2)	0.0198(5)
C4	0.9682(4)	0.25507(17)	0.6378(2)	0.0262(6)
H4	1.0005	0.2788	0.7133	0.031*
C5	0.9932(4)	0.16890(17)	0.6240(2)	0.0253(6)
H5	1.0408	0.1357	0.6916	0.030*
C6	0.8538(4)	0.39726(15)	0.5523(2)	0.0196(5)
C7	0.7504(4)	0.52277(16)	0.5234(2)	0.0207(5)
C8	0.6691(4)	0.60385(16)	0.4689(2)	0.0219(6)
C9	0.6096(4)	0.61492(17)	0.3476(2)	0.0285(6)
H9	0.6152	0.5697	0.2976	0.034*
C10	0.5419(4)	0.69349(19)	0.3011(3)	0.0338(7)
H10	0.5032	0.6994	0.2191	0.041*
C11	0.5868(5)	0.75032(19)	0.4824(3)	0.0390(8)
H11	0.5815	0.7970	0.5302	0.047*
C12	0.6545(5)	0.67443(18)	0.5369(3)	0.0350(7)
H12	0.6904	0.6703	0.6190	0.042*
O1	0.7502(3)	−0.00460(12)	0.36000(17)	0.0275(5)
H1A	0.7714	−0.0220	0.2937	0.041*

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Abstract

$C_{24}H_{28}O_6N_{10}Ni$, monoclinic, $P2_1/c$ (no. 14), $a = 7.3274(4)$ Å, $b = 15.6403(5)$ Å, $c = 11.8035(6)$ Å, $\beta = 106.966(5)^\circ$, $V = 1293.85(10)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0397$, $wR_{ref}(F^2) = 0.1002$, $T = 293(2)$ 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

A mixture of $Ni(OAc)_2 \cdot 4H_2O$ (0.1 mmol, 0.0248 g), THF (10 mL), 3,5-bis(4-pyridine)-1,2,4-triazole acid (0.2 mmol, 278 mg) (3,5-bpta) was dissolved in 8 mL H_2O . Then the solution was heated in a 23 mL Teflon-lined autoclave under

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
O2	0.8476(3)	−0.03241(13)	0.61879(18)	0.0264(4)
O3	0.6179(3)	0.07657(12)	0.70668(18)	0.0289(5)
H3A	0.6609	0.0822	0.7814	0.043*
H3B	0.6173	0.1270	0.6799	0.043*
H2A	0.918(6)	−0.051(2)	0.681(4)	0.050(11)*
H2B	0.760(6)	0.004(2)	0.639(3)	0.050(11)*
H1B	0.623(6)	−0.011(2)	0.351(3)	0.050(11)*

autogenous pressure at 393 K for five days. After cooling to room temperature green crystals formed.

Experimental details

Coordinates of hydrogen atoms were refined without any constraints or restraints. Their U_{iso} values were set to $1.2U_{eq}$ of the parent atoms.

Comment

The design and construction of metal-organic frameworks (MOFs) is the hottest research. The organic ligands, which are different in the size, the exhibility, the coordination ability, the number of the N-donor ligands, have great influence on structure of final product [4–6]. The flexibility of flexible N-donor ligands can result not only in various conformations in coordination, but also in free rotation to meet the requirements of the coordination geometry of the metal ions in the assembly process. We have already shown that some N-based molecules serve as bridging ligands to coordinate metal ions [7–9].

In the title complex, there are one half of a Ni(II) ion, one 3,5-bpta ligand, and three water molecules in the asymmetric unit. The nickel atom Ni1 is six-coordinated with a distorted octahedral geometry by two N atoms from the 3,5-bpta ligands and four water molecules (see the figure). The Ni–N bond length is 2.092(2) Å; and the Ni–O ones are in the range of 2.0768(18) to 2.0916(19) Å, respectively. All geometric parameters are comparable to related ones [10–12]. The hydrogen bonding between carboxylate oxygen atoms and the NH group of the organic ligands are: d(O···N): 2.936(3) Å and N–H···O is 174.7°.

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