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Crystal structure of the nickel(II) complex aqua-(2,6-di(pyrazin-2-yl)-4,4'-bipyridine- $\kappa^3 N, N', N''$)-(phthalato- $\kappa^2 O, O'$)nickel(II) tetrahydrate, $C_{26}H_{26}N_6O_9Ni$

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Abstract

$C_{26}H_{26}N_6O_9Ni$, triclinic, $P\bar{1}$ (no. 2), $a = 11.2329(5)$ Å, $b = 11.7535(5)$ Å, $c = 12.1109(7)$ Å, $\alpha = 70.936(5)^\circ$, $\beta = 75.684(4)^\circ$, $\gamma = 63.266(4)^\circ$, $V = 1340.13(13)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0314$, $wR_{ref}(F^2) = 0.0743$, $T = 290$ 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.

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Table 1: Data collection and handling.

Crystal:	Green block
Size:	0.18 × 0.18 × 0.18 mm
Wavelength:	MoK α radiation (0.71073 Å)
μ :	0.79 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
θ_{max} , completeness:	25.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	25097, 4994, 0.033
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 4479
$N(param)_{refined}$:	383
Programs:	CrysAlis ^{PRO} [1], Olex2 [2], SHELX [3]

Source of material

A mixture of 2,6-di(pyrazin-2-yl)-4,4'-bipyridine (py-pzppyz; 0.1 mmol), phthalic acid (0.1 mmol), $Ni(OAc)_2 \cdot 4H_2O$ (0.1 mmol) and H_2O (10 mL) was stirred for 30 min, and the pH value of the solution was adjusted to about 4 with 1 M KOH. Then the mixture was transferred to a 25 mL Teflon-lined stainless steel vessel and heated at 140 °C for three days, and then cooled to room temperature over 40 h. Green block crystals of the title compound were obtained. **Anal. Calcd.** for $C_{26}H_{26}NiN_6O_9$ (%): C, 49.95, H 4.19, N 13.44. Found: C 49.76, H 4.21, N 13.40.

Experimental details

Using Olex2, the structure was solved with the SIR2004 structure solution program using Direct Methods and refined with the ShelXL refinement package using least squares minimisation.

Comment

Coordination compounds or frameworks have been receiving extensive attention not only for their aesthetics of structures but also for their potential applications, such as photoluminescence, gas storage, catalysis, etc. [4–6]. The construction of the compounds involve a variety of

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	0.35072 (2)	0.12411 (2)	0.20228 (2)	0.02452 (8)
O1	0.29144 (14)	0.00449 (14)	0.34780 (12)	0.0329 (3)
O2	0.19155 (15)	−0.13132 (15)	0.43954 (13)	0.0397 (4)
O3	0.37212 (14)	0.00281 (15)	0.10788 (14)	0.0390 (4)
O4	0.33128 (15)	−0.13947 (14)	0.05772 (13)	0.0374 (4)
O5	0.40718 (15)	0.23714 (15)	0.04638 (13)	0.0414 (4)
H5A	0.4926	0.1996	0.0246	0.062*
H5B	0.3903	0.3138	0.0548	0.062*
N1	0.36508 (15)	0.21314 (15)	0.31269 (14)	0.0246 (4)
N2	0.55193 (16)	0.01222 (15)	0.24189 (15)	0.0272 (4)
N3	0.79606 (17)	−0.13547 (17)	0.33220 (17)	0.0379 (4)
N4	0.15872 (16)	0.29271 (15)	0.20248 (14)	0.0255 (4)
N5	−0.06954 (18)	0.52281 (19)	0.21721 (19)	0.0448 (5)
N6	0.4074 (2)	0.51259 (19)	0.70930 (17)	0.0420 (5)
C25	0.10765 (19)	0.02286 (18)	0.26608 (16)	0.0238 (4)
C1	0.26134 (19)	0.31988 (18)	0.33826 (17)	0.0253 (4)
C2	0.2670 (2)	0.3784 (2)	0.41770 (18)	0.0295 (5)
H2	0.1944	0.4531	0.4342	0.035*
C3	0.38219 (19)	0.32516 (19)	0.47303 (17)	0.0268 (4)
C4	0.48814 (19)	0.21200 (19)	0.44689 (17)	0.0270 (4)
H4	0.5657	0.1727	0.4836	0.032*
C5	0.47601 (18)	0.15946 (18)	0.36584 (17)	0.0247 (4)
C6	0.58125 (18)	0.04232 (18)	0.32681 (17)	0.0248 (4)
C7	0.7028 (2)	−0.0332 (2)	0.37236 (19)	0.0318 (5)
H7	0.7197	−0.0119	0.4327	0.038*
C8	0.7670 (2)	−0.1607 (2)	0.2453 (2)	0.0393 (5)
H8	0.8311	−0.2299	0.2137	0.047*
C9	0.6455 (2)	−0.0885 (2)	0.19992 (19)	0.0335 (5)
H9	0.6289	−0.1104	0.1397	0.040*
C11	0.14592 (19)	0.36643 (19)	0.27284 (17)	0.0265 (4)
C10	0.0318 (2)	0.4803 (2)	0.2789 (2)	0.0401 (5)
H10	0.0256	0.5293	0.3284	0.048*
C13	−0.0555 (2)	0.4492 (2)	0.1484 (2)	0.0381 (5)
H13	−0.1242	0.4750	0.1040	0.046*
C12	0.0575 (2)	0.3355 (2)	0.14030 (18)	0.0315 (5)
H12	0.0632	0.2875	0.0901	0.038*
C14	0.39157 (19)	0.38853 (19)	0.55568 (17)	0.0268 (4)
C18	0.3224 (2)	0.5229 (2)	0.54338 (19)	0.0340 (5)
H18	0.2690	0.5743	0.4828	0.041*
C17	0.3332 (2)	0.5794 (2)	0.6209 (2)	0.0389 (5)
H17	0.2859	0.6697	0.6110	0.047*
C16	0.4742 (2)	0.3831 (2)	0.7211 (2)	0.0417 (6)
H16	0.5266	0.3344	0.7827	0.050*
C15	0.4701 (2)	0.3177 (2)	0.64765 (18)	0.0341 (5)
H15	0.5190	0.2273	0.6593	0.041*
C20	0.14942 (19)	0.01603 (18)	0.14890 (17)	0.0253 (4)
C21	0.0533 (2)	0.0687 (2)	0.07194 (19)	0.0338 (5)
H21	0.0800	0.0608	−0.0052	0.041*
C22	−0.0813 (2)	0.1327 (2)	0.1085 (2)	0.0384 (5)
H22	−0.1444	0.1696	0.0557	0.046*
C23	−0.1216 (2)	0.1414 (2)	0.2235 (2)	0.0365 (5)
H23	−0.2118	0.1859	0.2480	0.044*
C24	−0.0285 (2)	0.0843 (2)	0.30248 (19)	0.0322 (5)
H24	−0.0571	0.0870	0.3809	0.039*
C26	0.20419 (19)	−0.03962 (19)	0.35746 (16)	0.0259 (4)

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C19	0.2951 (2)	−0.04567 (19)	0.10260 (16)	0.0270 (4)
O7	0.3033 (2)	0.50275 (19)	1.04469 (19)	0.0786 (7)
H7A	0.2585	0.5554	1.0887	0.118*
H7B	0.3462	0.5404	0.9898	0.118*
O8	0.2045 (3)	0.6817 (2)	1.1769 (2)	0.0865 (8)
H8A	0.1639	0.6998	1.2429	0.097 (13)*
H8B	0.2437	0.7274	1.1407	0.105 (15)*
O6	0.4292 (2)	0.63299 (19)	0.85914 (16)	0.0630 (5)
H6A	0.4164	0.6019	0.8109	0.095*
H6B	0.5095	0.5898	0.8756	0.095*
O9	0.06329 (17)	0.72532 (18)	1.39954 (17)	0.0593 (5)
H9A	−0.0184	0.7705	1.3875	0.086 (11)*
H9B	0.0806	0.7784	1.4169	0.088 (12)*

building blocks (metal ions, organic ligands, counter anions, solvents, etc.) [7, 8]. The carboxylate ligands have been extensively employed due to their versatile coordination conformations and strong coordination ability. Moreover, carboxylate ligands are usually utilized as hydrophilic groups to form hydrogen bonds. Hydrogen bonds often play a dominant role in crystal engineering because of their selectivity and directionality to control the design in the synthesis of assemblies that contain N-based ligands and carboxylates [9]. The pyrazine analogues ligands have received some attention on account of the weaker σ -donor and stronger π -acceptor nature of pyrazine [10, 11]. This contribution is part of a study on 4-(4-pyridyl)-2,5-dipyrzylpyridine [12–14]. The interesting feature of the ligand is that it is able to a chelating coordination and a monodentate one.

The ORPEP-type drawing of the title compound is shown in the Figure. The structure consists of the title complex and four isolated water molecules. The center Ni(II) ion resides in a distorted octahedron center and is coordinated by three nitrogen atoms from a py-pzpypz ligand and two oxygen atoms from the phthalate ligand and one coordinated water molecular. The equatorial plane is composed of three nitrogen atoms and one oxygen atom (N1, N2, N4, O3), while atoms O1 and O5 occupy the axial positions. The average Ni–N bond length is 2.1071(16) Å, and the Ni–O bond lengths are in a range of 2.0045(14)–2.0638(15) Å, which are comparable to those found in other reported Ni(II) compounds [15, 16].

The intermolecular hydrogen bonds occur between two oxygen atoms from coordinated water molecules and non-coordinated water molecules and between uncoordinated water molecule. The intermolecular hydrogen bonds also occur between two oxygen atoms from

coordinated water molecule and one carboxylate group and between two oxygen atoms from non-coordinated water molecule and a carboxylate group. Thereby, the title compound can be viewed as a three-dimensional architecture extended via these hydrogen bonds.

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