Crystal structure of *di*-aqua-*bis*-(2-(4-methyl-5-oxo-4-propan-2-yl-1*H*-imidazol-2-yl)pyridine-3-carboxylic acid-nickel(II), $Ni(C_{13}H_{14}N_3O_3)_2(H_2O)_2$, $C_{26}H_{32}N_6NiO_8$

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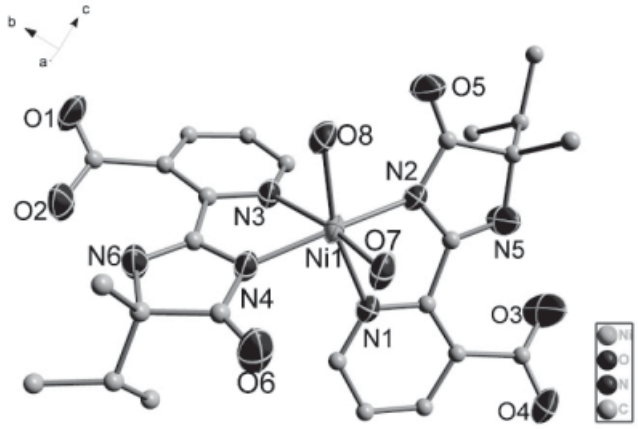

Abstract

$C_{26}H_{32}N_6NiO_8$, orthorhombic, *Pbca* (no. 61), a = 12.511(3) Å, b = 19.521(5) Å, c = 23.053(6) Å, V = 5630.2 Å^3, Z = 8, $R_{gt}(F)$ = 0.0541, $wR_{ref}(F^2)$ = 0.1570, T = 296 K.

Table 1. Data collection and handling.

Crystal:	dark green blocks, size 0.21×0.17×0.15 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	7.48 cm^{-1}
Diffractometer, scan mode:	CCD area detector, ω and φ
$2\theta_{max}$:	56.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	34797, 6687
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4864
$N(param)_{refined}$:	384
Programs:	SHELX [2]

Source of material

A mixture of nickel acetate (0.5 mmol), *imazapyr*H (2-(4-methyl-5-oxo-4-propan-2-yl-1*H*-imidazol-2-yl)pyridine-3-carboxylic acid, $C_{13}H_{15}N_3O_3$, 0.5 mmol), H_2O (10 mL) and alcohol (10 mL) was stirred for 30 min, and then the pH value was adjusted to 6 with a solution of NaOH. After stirring for 1 h, the mixture was transferred to a 30 mL Teflon-lined stainless steel reactor and kept at 413 K under autogenous pressure for 3 days. The reactor was cooled to room temperature at a rate of 10K per hour. Crystals suitable for X-ray diffraction were obtained and washed repeatedly with distilled water and alcohol.

Elemental analysis for $C_{26}H_{32}N_6O_8Ni$-found(%): C, 49.85; H, 4.48; N, 14.20; O, 21.67. calcd(%): C, 50.05; H, 4.83; N, 14.00; O, 21.32.

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Discussion

The title mononuclear nickel complex, $C_{26}H_{32}N_6O_8Ni$, is a hydrogen bonded polymer. The asymmetric unit consists of one Ni atom, two *imazapyr* ligands and two coordinated water molecules. The Ni atom is sixfold-coordinated by four N atoms from two *imazapyr* and two O atoms from two coordinated water molecules, in a distorted octahedral manner. In each complex, there are not only two intramolecular O–H···O hydrogen bonds between the coordinated water molecules and the carbonyl O atoms, but also two intramolecular N–H···O hydrogen bonds between the carboxylate group of the *imazapyr* and the neighboring H atom of the imidazole ring. The hydrogen-acceptor bond length are with 2.0(1) Å for O-H···O, 1.5(1) Å and 1.6(1) for N-H···O type hydrogen bonds in the typical range. In addition to this, there are two intermolecular symmetry related O–H···O hydrogen bonds between adjacent molecules, which show a H···O distance of 1.83(1) Å. Hence, the complexes are linked to 1D chains by the strong intermolecular hydrogen bonding interaction.

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

Atom	Site	x	y	z	U_{iso}
H(7C)	8*c*	0.1058	0.0076	0.3412	0.054
H(7D)	8*c*	0.1171	0.0609	0.3021	0.054
H(8C)	8*c*	0.1114	0.2121	0.4103	0.052
H(8D)	8*c*	0.1243	0.1597	0.4500	0.052
H(28)	8*c*	0.317(4)	–0.093(3)	0.481(2)	0.09(2)
H(29)	8*c*	0.323(5)	0.314(3)	0.266(2)	0.11(2)
H(1A)	8*c*	0.3484	0.3154	0.1533	0.156
H(1B)	8*c*	0.2444	0.3050	0.1164	0.156
H(1C)	8*c*	0.3528	0.2732	0.0954	0.156
H(2)	8*c*	0.2430	0.1891	0.1368	0.078
H(3A)	8*c*	0.4316	0.2068	0.2013	0.102
H(3B)	8*c*	0.4237	0.1678	0.1420	0.102
H(3C)	8*c*	0.3681	0.1377	0.1974	0.102
H(5A)	8*c*	0.0705	0.2723	0.2375	0.103
H(5B)	8*c*	0.0751	0.2469	0.1730	0.103
H(5C)	8*c*	0.1304	0.3159	0.1905	0.103
H(11)	8*c*	0.4822	0.3441	0.4412	0.055
H(12)	8*c*	0.4918	0.2465	0.4977	0.070
H(13)	8*c*	0.3964	0.1512	0.4701	0.059
H(16A)	8*c*	0.0697	–0.0600	0.5019	0.107
H(16B)	8*c*	0.0589	–0.0289	0.5643	0.107
H(16C)	8*c*	0.1159	–0.0996	0.5555	0.107
H(17)	8*c*	0.2912	–0.0627	0.5965	0.121
H(22)	8*c*	0.4693	–0.1283	0.3018	0.053
H(23)	8*c*	0.4725	–0.0320	0.2441	0.062
H(24)	8*c*	0.3869	0.0655	0.2756	0.059
H(25A)	8*c*	0.3825	0.0359	0.5377	0.175
H(25B)	8*c*	0.4350	–0.0087	0.5866	0.175
H(25C)	8*c*	0.3791	0.0607	0.6024	0.175
H(26A)	8*c*	0.2517	0.0168	0.6705	0.184
H(26B)	8*c*	0.1465	–0.0122	0.6429	0.184
H(26C)	8*c*	0.1870	0.0612	0.6261	0.184

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*	0.22780(3)	0.10910(2)	0.37479(2)	0.0380(2)	0.0171(2)	0.0356(2)	0.0002(1)	–0.0002(2)	0.0038(1)
O(1)	8*c*	0.4030(3)	0.4231(1)	0.3748(1)	0.098(2)	0.022(1)	0.066(2)	0.002(1)	–0.014(2)	–0.003(1)
O(2)	8*c*	0.3810(3)	0.3732(1)	0.2904(1)	0.098(2)	0.037(1)	0.051(2)	–0.025(1)	0.002(1)	0.009(1)
O(3)	8*c*	0.3878(4)	–0.1539(2)	0.4559(1)	0.192(4)	0.056(2)	0.056(2)	0.068(2)	0.006(2)	0.016(1)
O(4)	8*c*	0.3986(3)	–0.2058(1)	0.3728(1)	0.090(2)	0.023(1)	0.078(2)	–0.002(1)	0.009(2)	0.004(1)
O(5)	8*c*	0.1502(3)	0.1022(1)	0.5230(1)	0.094(2)	0.048(1)	0.047(1)	0.027(1)	0.021(1)	0.003(1)
O(6)	8*c*	0.1411(3)	0.1166(1)	0.2275(1)	0.101(2)	0.051(2)	0.052(2)	–0.030(2)	–0.025(2)	0.005(1)
O(7)	8*c*	0.1056(2)	0.0510(1)	0.3375(1)	0.055(1)	0.025(1)	0.056(1)	–0.0095(9)	–0.008(1)	0.0046(9)
O(8)	8*c*	0.1107(2)	0.1689(1)	0.4147(1)	0.051(1)	0.025(1)	0.053(1)	0.0088(9)	0.005(1)	0.0056(9)
N(1)	8*c*	0.3368(2)	0.0298(1)	0.3498(1)	0.045(1)	0.025(1)	0.039(1)	0.005(1)	0.002(1)	0.004(1)
N(2)	8*c*	0.2376(2)	0.0509(1)	0.4464(1)	0.044(1)	0.022(1)	0.036(1)	0.005(1)	0.004(1)	0.002(1)
N(3)	8*c*	0.3415(2)	0.1873(1)	0.3972(1)	0.041(1)	0.025(1)	0.038(1)	–0.002(1)	–0.005(1)	0.005(1)
N(4)	8*c*	0.2338(2)	0.1669(1)	0.3027(1)	0.046(1)	0.022(1)	0.036(1)	–0.005(1)	–0.005(1)	0.003(1)
N(5)	8*c*	0.2774(3)	–0.0501(2)	0.4855(1)	0.111(3)	0.046(2)	0.039(2)	0.037(2)	0.013(2)	0.013(1)
N(6)	8*c*	0.2800(3)	0.2661(1)	0.2613(1)	0.074(2)	0.033(1)	0.039(2)	–0.014(1)	–0.006(1)	0.007(1)
C(1)	8*c*	0.3100(6)	0.2842(3)	0.1288(2)	0.139(5)	0.095(4)	0.077(4)	–0.022(4)	0.021(4)	0.029(3)
C(2)	8*c*	0.2852(4)	0.2184(2)	0.1626(2)	0.081(3)	0.065(3)	0.048(2)	–0.012(2)	0.003(2)	0.002(2)
C(3)	8*c*	0.3864(4)	0.1790(2)	0.1772(2)	0.068(3)	0.074(3)	0.063(3)	0.010(2)	0.018(2)	–0.002(2)
C(4)	8*c*	0.2171(3)	0.2315(2)	0.2162(2)	0.062(2)	0.043(2)	0.048(2)	–0.007(2)	–0.009(2)	0.007(2)
C(5)	8*c*	0.1137(3)	0.2702(2)	0.2031(2)	0.061(2)	0.073(3)	0.072(3)	0.016(2)	–0.015(2)	0.016(2)
C(6)	8*c*	0.1923(3)	0.1640(2)	0.2476(2)	0.063(2)	0.037(2)	0.044(2)	–0.011(2)	–0.009(2)	0.004(1)
C(7)	8*c*	0.2842(2)	0.2273(1)	0.3073(1)	0.045(2)	0.025(1)	0.035(2)	–0.002(1)	0.000(1)	0.003(1)
C(8)	8*c*	0.3417(2)	0.2427(1)	0.3620(1)	0.031(1)	0.022(1)	0.036(2)	0.000(1)	0.002(1)	0.001(1)
C(9)	8*c*	0.3910(2)	0.3042(1)	0.3773(1)	0.033(1)	0.023(1)	0.040(2)	–0.001(1)	0.002(1)	–0.002(1)
C(10)	8*c*	0.3907(2)	0.3723(1)	0.3440(2)	0.035(2)	0.025(1)	0.052(2)	–0.003(1)	–0.001(1)	0.003(1)
C(11)	8*c*	0.4473(3)	0.3044(2)	0.4294(2)	0.051(2)	0.031(2)	0.056(2)	–0.008(1)	–0.012(2)	–0.004(1)
C(12)	8*c*	0.4519(3)	0.2468(2)	0.4637(2)	0.073(3)	0.044(2)	0.057(2)	–0.010(2)	–0.033(2)	0.006(2)
C(13)	8*c*	0.3959(3)	0.1897(2)	0.4463(2)	0.062(2)	0.034(2)	0.052(2)	–0.006(2)	–0.019(2)	0.013(1)
C(14)	8*c*	0.1966(3)	0.0533(2)	0.5019(2)	0.067(2)	0.040(2)	0.040(2)	0.015(2)	0.008(2)	0.005(1)
C(15)	8*c*	0.2128(4)	–0.0177(2)	0.5312(2)	0.095(3)	0.050(2)	0.043(2)	0.021(2)	0.007(2)	0.007(2)
C(16)	8*c*	0.1039(4)	–0.0551(2)	0.5390(2)	0.083(3)	0.065(3)	0.066(3)	–0.019(2)	0.017(2)	0.014(2)
C(17)	8*c*	0.2740(5)	–0.0153(3)	0.5859(2)	0.137(5)	0.109(5)	0.057(3)	0.051(4)	–0.010(3)	0.002(3)
C(18)	8*c*	0.2846(3)	–0.0102(2)	0.4407(1)	0.048(2)	0.027(1)	0.037(2)	0.010(1)	–0.001(1)	0.003(1)
C(19)	8*c*	0.3392(2)	–0.0257(1)	0.3852(1)	0.036(2)	0.024(1)	0.037(2)	0.003(1)	–0.002(1)	0.002(1)
C(20)	8*c*	0.3869(2)	–0.0872(1)	0.3686(1)	0.039(2)	0.025(1)	0.042(2)	0.004(1)	–0.004(1)	0.000(1)
C(21)	8*c*	0.3904(3)	–0.1546(2)	0.4027(2)	0.049(2)	0.030(2)	0.055(2)	0.012(1)	0.003(2)	0.008(1)
C(22)	8*c*	0.4365(3)	–0.0882(2)	0.3145(2)	0.052(2)	0.029(1)	0.051(2)	0.008(1)	0.003(2)	–0.007(1)
C(23)	8*c*	0.4379(3)	–0.0314(2)	0.2798(2)	0.066(2)	0.045(2)	0.044(2)	0.007(2)	0.017(2)	0.002(2)
C(24)	8*c*	0.3866(3)	0.0268(2)	0.2991(2)	0.065(2)	0.035(2)	0.047(2)	0.010(2)	0.014(2)	0.011(1)
C(25)	8*c*	0.3767(5)	0.0213(4)	0.5774(3)	0.076(4)	0.191(7)	0.083(4)	–0.033(4)	–0.018(3)	–0.022(4)
C(26)	8*c*	0.2087(7)	0.0155(4)	0.6360(2)	0.203(8)	0.122(5)	0.043(3)	0.047(5)	0.015(4)	–0.002(3)

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