Crystal structure of tetraaquabis(4-nitropyridine *N*-oxide-*O*)nickel(II) dinitrate, $[\text{Ni}(\text{H}_2\text{O})_4(\text{ONC}_5\text{H}_4\text{NO}_2)_2][\text{NO}_3]_2$

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Abstract

$\text{C}_{10}\text{H}_{16}\text{N}_6\text{NiO}_{16}$, monoclinic, $P12_1/c1$ (no. 14), $a = 8.332(2)$ Å, $b = 11.336(2)$ Å, $c = 11.087(2)$ Å, $\beta = 102.430(3)^\circ$, $V = 1022.7$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.042$, $wR_{\text{ref}}(F^2) = 0.095$, $T = 298$ K.

Source of material

4-Nitropyridine *N*-oxide (0.0623 g, 0.445 mmol, in 15 ml CHCl_3) was added to a 15 ml methanol solution containing $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (0.0754 g, 0.259 mmol) and the solution was stirred for a few minutes. The yellow single crystals were obtained after the solution was allowed to stand at room temperature for slow evaporation of the solvents about four weeks.

Discussion

Pyridine *N*-oxide and its derivatives are a kind of useful ligands and several complexes have been synthesized with these compounds as bridging or terminal ligands [1,2]. They are of interest

for understanding the relationship between coordination modes and metallic ions.

In the crystal structure of the title complex, the Ni^{II} atom possesses a distorted NiO_6 octahedral environment, in which two 4-nitropyridine *N*-oxide ligands are *trans*-coordinated at the Ni^{II} atom via the terminal O atoms. The other four coordinating O atoms come from four water molecules. The Ni^{II} atom is located on an inversion center. The coordination fashion of this complex is identical with that of the Co^{II} complex $[\text{Co}(\text{C}_5\text{H}_4\text{N}_2\text{O}_3)(\text{H}_2\text{O})_4](\text{NO}_3)_2$ [3]. There exist hydrogen bonds between the coordinating water molecules and nitrate anions, and in addition, there are a very weak π - π stacking interactions between neighboring pyridine rings. The relevant distances are $d(\text{Cg1} \cdots \text{Cg1}^{\text{I}}) = 5.563(2)$ Å, $d(\text{Cg1} \cdots \text{Rp1}^{\text{I}}_{\text{perp}}) = 3.037$ Å, $d(\text{Cg1} \cdots \text{Cg1}^{\text{II}}) = 5.563(2)$ Å and $d(\text{Cg1} \cdots \text{Rp1}^{\text{II}}_{\text{perp}}) = 3.070$ Å [symmetry codes: (i) $x, \frac{1}{2}-y, -\frac{1}{2}+z$; (ii) $x, \frac{1}{2}-y, \frac{1}{2}+z$; Cg1 is the centroid of the N1/C1-C5 ring; $d(\text{Cg1}^{\text{I}} \cdots \text{Rp1}^{\text{J}}_{\text{perp}})$ is the perpendicular distance from Cg1^{I} to plane of ring J]. The hydrogen bonds and the π - π stacking interaction make the connections between the coordinated cations and the counter anions and which led to the formation of the three-dimensional network.

Table 1. Data collection and handling.

Crystal:	yellow prism, size 0.20 × 0.23 × 0.34 mm
Wavelength:	Mo K_{α} radiation (0.71073 Å)
μ :	10.43 cm ⁻¹
Diffractometer, scan mode:	Bruker SMART APEX CCD, φ/ω
$2\theta_{\text{max}}$:	54°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	5871, 2197
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1982
$N(\text{param})_{\text{refined}}$:	151
Program:	SHELXTL [4]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(6)	4e	0.3526	0.3619	0.6206	0.063
H(5)	4e	0.3140	0.3229	0.4976	0.063
H(7)	4e	0.4975	0.5367	0.7324	0.068
H(8)	4e	0.5739	0.6385	0.6956	0.068
H(2)	4e	0.9603	0.3019	0.2945	0.057
H(3)	4e	0.7774	0.0292	0.4512	0.053
H(1)	4e	0.8443	0.4286	0.4136	0.053
H(4)	4e	0.6628	0.1601	0.5667	0.048

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Ni(1)	2b	½	½	½	0.0395(3)	0.0230(2)	0.0316(2)	0.0010(2)	0.0115(2)	−0.0023(2)
O(7)	4e	0.3255(2)	0.3878(2)	0.5432(2)	0.053(1)	0.0336(9)	0.043(1)	−0.0048(8)	0.0183(9)	−0.0013(7)
O(3)	4e	0.6769(2)	0.3766(2)	0.5689(2)	0.059(1)	0.041(1)	0.041(1)	0.0198(9)	0.0151(9)	0.0023(8)
O(4)	4e	0.3966(3)	0.1989(2)	0.2837(2)	0.099(2)	0.033(1)	0.050(1)	−0.017(1)	0.027(1)	−0.0029(9)
N(2)	4e	0.3567(3)	0.1270(2)	0.3572(2)	0.054(1)	0.035(1)	0.041(1)	−0.005(1)	0.014(1)	−0.0046(9)
O(8)	4e	0.5449(2)	0.5651(2)	0.6744(2)	0.069(1)	0.0336(9)	0.0363(9)	−0.0098(9)	0.0191(9)	−0.0086(7)
N(1)	4e	0.7424(3)	0.3054(2)	0.4990(2)	0.037(1)	0.034(1)	0.036(1)	0.0054(9)	0.0097(9)	0.0035(8)
O(5)	4e	0.3003(3)	0.1614(2)	0.4454(2)	0.086(2)	0.046(1)	0.056(1)	−0.006(1)	0.034(1)	−0.014(1)
O(6)	4e	0.3738(3)	0.0200(2)	0.3406(2)	0.092(2)	0.0284(9)	0.068(1)	0.002(1)	0.043(1)	−0.0016(9)
C(4)	4e	0.8995(4)	0.2728(3)	0.3492(3)	0.046(2)	0.052(2)	0.050(2)	0.002(1)	0.021(1)	0.010(1)
C(1)	4e	0.7903(3)	0.1101(2)	0.4426(3)	0.048(2)	0.030(1)	0.055(2)	0.005(1)	0.010(1)	0.003(1)
N(3)	4e	0.9503(3)	0.0706(3)	0.2858(2)	0.048(2)	0.068(2)	0.052(1)	0.019(1)	0.003(1)	−0.012(1)
C(3)	4e	0.8304(3)	0.3476(2)	0.4200(3)	0.048(2)	0.032(1)	0.055(2)	0.001(1)	0.019(1)	0.008(1)
O(2)	4e	1.0193(3)	0.1119(3)	0.2084(2)	0.056(1)	0.110(2)	0.057(1)	0.017(1)	0.020(1)	−0.015(1)
O(1)	4e	0.9380(4)	−0.0343(2)	0.3065(3)	0.107(2)	0.059(2)	0.098(2)	0.032(2)	0.027(2)	−0.018(1)
C(2)	4e	0.7229(3)	0.1880(2)	0.5110(2)	0.042(1)	0.037(1)	0.045(1)	0.001(1)	0.014(1)	0.010(1)
C(5)	4e	0.8774(3)	0.1534(2)	0.3606(2)	0.038(1)	0.045(1)	0.041(1)	0.011(1)	0.007(1)	−0.003(1)

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