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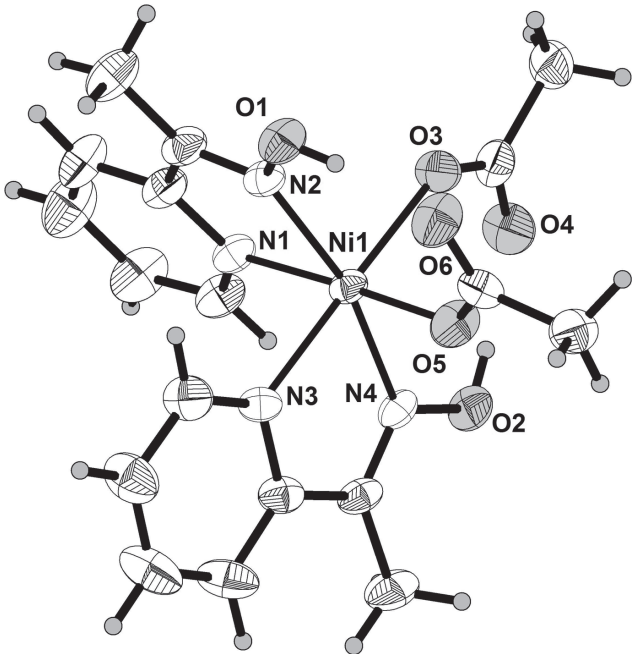
Crystal structure of bis(acetato-κ*O*)-bis(1-(pyridin-2-yl)ethan-1-one oxime-κ²*N*,*N*′)nickel(II), $C_{18}H_{22}N_4NiO_6$



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Abstract

$C_{18}H_{22}N_4NiO_6$, triclinic, $P\bar{1}$ (no. 2), $a = 9.3398(13)$ Å, $b = 9.9707(11)$ Å, $c = 12.0063(18)$ Å, $\alpha = 89.829(3)°$, $\beta = 71.931(2)°$, $\gamma = 74.243(2)°$, $V = 1019.0(2)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0472$, $wR_{ref}(F^2) = 0.1305$, $T = 298(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

Hydroxylamine hydrochloride (0.695 g, 10 mmol) was added to sodium hydroxide (0.401 g, 10 mmol) in deionised water (10 mL). The solution was then added dropwise to 2-acetylpyridine (1.211 g, 10 mmol) in methanol (10 mL). The reaction was stirred for 3 h. A solution of nickel acetate tetrahydrate (2.488 g, 10 mmol) in methanol (10 mL) was added. The resulting green solution was stirred at 25 °C for about 6 h and was then allowed to slowly concentrate by solvent evaporation at room temperature. Green block crystals were obtained within 2 weeks. Elemental analysis calculated for $C_{18}H_{22}N_4NiO_6$: C 48.14, H 4.94, N 12.48%; found: C 47.87, H 5.07, N 12.23%.

Table 1: Data collection and handling.

Crystal:	Green block
Size:	0.40 × 0.25 × 0.21 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	9.9 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	50°, 98.5%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5320, 3535, 0.019
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2726
$N(param)_{refined}$:	262
Programs:	Bruker programs [1], SHELX [2]

Experimental details

All hydrogen atoms were placed geometrically and treated as riding on their parent atoms with O—H 0.82 Å, C—H 0.96 Å [U_{iso}(H) = 1.5 times U_{eq} (O, water), (C, methyl)] and C—H 0.93 Å [U_{iso}(H) = 1.2 times U_{eq} (C, aromatic ring)].

Comment

There is currently a renewed interest in the coordination chemistry of oximes and their application of metal ion/oxime systems as simple and efficient catalysts and their use in synthesis of coordination polymers with interesting magnetic properties [3–5]. In the area of molecular magnetism, the oximato group of the anionic pyridyl oximes can mediate exchange interactions of varying range, from weak ferromagnetic to strong antiferromagnetic [6, 7].

The title complex consists of nickel, two oxime and two acetate ligands. The Ni(II) ion is hexa-coordinated in a distorted octahedron coordination geometry, defined by two O

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.59354(5)	0.73147(5)	0.73138(4)	0.03536(19)
N1	0.4185(4)	0.9162(3)	0.8124(3)	0.0417(8)
N2	0.5667(4)	0.7166(3)	0.9081(3)	0.0405(7)
N3	0.4157(4)	0.6373(3)	0.7391(3)	0.0395(7)
N4	0.5596(4)	0.7624(3)	0.5691(3)	0.0393(7)
O1	0.6463(3)	0.6084(3)	0.9558(2)	0.0537(7)
H1	0.7146	0.5533	0.9029	0.081*
O2	0.6368(3)	0.8313(3)	0.4823(2)	0.0529(7)
H2	0.6903	0.8686	0.5073	0.079*
O3	0.7737(3)	0.8156(3)	0.7225(2)	0.0545(7)
O4	0.7903(4)	0.9425(3)	0.5696(3)	0.0666(9)
O5	0.7553(3)	0.5448(3)	0.6544(2)	0.0532(7)
O6	0.8373(4)	0.4357(3)	0.7933(3)	0.0694(9)
C1	0.4202(6)	0.8047(5)	1.1140(4)	0.0647(13)
H1A	0.3371	0.8865	1.1532	0.097*
H1B	0.3867	0.7227	1.1357	0.097*
H1C	0.5111	0.8009	1.1366	0.097*
C2	0.4604(5)	0.8110(4)	0.9835(3)	0.0433(9)
C3	0.3734(5)	0.9268(4)	0.9314(3)	0.0440(9)
C4	0.2555(6)	1.0398(5)	0.9975(4)	0.0678(14)
H4	0.2286	1.0474	1.0791	0.081*
C5	0.1776(7)	1.1418(5)	0.9414(5)	0.0827(17)
H5	0.0966	1.2179	0.9847	0.099*
C6	0.2221(6)	1.1287(5)	0.8201(5)	0.0731(15)
H6	0.1702	1.1952	0.7807	0.088*
C7	0.3424(5)	1.0175(4)	0.7590(4)	0.0530(11)
H7	0.3736	1.0110	0.6772	0.064*
C8	0.4286(6)	0.7247(5)	0.4293(4)	0.0618(12)
H8A	0.3500	0.6794	0.4294	0.093*
H8B	0.3920	0.8219	0.4180	0.093*
H8C	0.5237	0.6813	0.3666	0.093*
C9	0.4598(5)	0.7116(4)	0.5443(3)	0.0422(9)
C10	0.3759(5)	0.6408(4)	0.6400(4)	0.0445(10)
C11	0.2586(6)	0.5852(5)	0.6333(5)	0.0694(14)
H11	0.2323	0.5868	0.5646	0.083*
C12	0.1803(6)	0.5271(6)	0.7298(6)	0.0816(17)
H12	0.0994	0.4908	0.7271	0.098*
C13	0.2231(6)	0.5234(5)	0.8291(5)	0.0707(14)
H13	0.1722	0.4846	0.8949	0.085*
C14	0.3420(5)	0.5779(4)	0.8295(4)	0.0508(10)
H14	0.3728	0.5731	0.8963	0.061*
C15	0.8333(5)	0.8940(4)	0.6534(4)	0.0453(10)
C16	0.9681(5)	0.9370(5)	0.6739(4)	0.0621(12)
H16A	0.9911	0.8937	0.7403	0.093*
H16B	1.0594	0.9076	0.6053	0.093*
H16C	0.9388	1.0369	0.6890	0.093*
C17	0.8409(4)	0.4466(4)	0.6893(4)	0.0446(9)
C18	0.9607(5)	0.3351(5)	0.5987(4)	0.0610(12)
H18A	0.9518	0.3551	0.5224	0.091*
H18B	1.0641	0.3328	0.5988	0.091*
H18C	0.9429	0.2459	0.6168	0.091*

atoms from the acetate ligands and four N atoms from the oxime ligands. The basal plane is formed by the atoms N1, N2, N4 and 05, the axial position of the octahedron is occupied by N3 and O3. The Ni—N bond lengths range from 2.068(3) Å to 2.098(3) Å and the Ni—O bond lengths are 2.049(3) Å, and 2.054(3) Å respectively. The bond angles surrounding the Ni atom are in the range of 76.67(12)° to 177.68(12)°. Bond lengths and angles are in the expected ranges [8–12].

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