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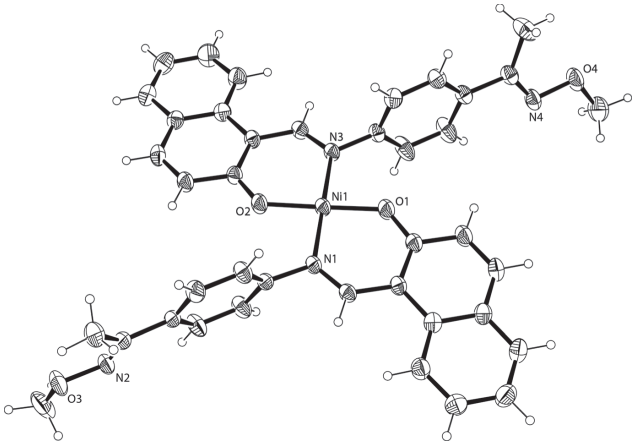
Synthesis and crystal structure of bis{((*E*)-((4-((*E*)-1-(methoxyimino)ethyl)phenyl)imino)methyl)-2-naphtholato-κ²*N*,*O*}nickel(II), $C_{40}H_{34}N_4NiO_4$



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Abstract

$C_{40}H_{34}N_4NiO_4$, monoclinic, $P2_1/c$ (no. 14), $a = 21.443(7)$ Å, $b = 13.862(5)$ Å, $c = 11.733(4)$ Å, $\beta = 103.690(5)°$, $Z = 4$, $V = 3388(2)$ Å^3, $R_{gt}(F) = 0.0484$, $wR_{ref}(F^2) = 0.1411$, $T = 296(2)$ K.

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Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

Synthesis of the ligand: All solvents and other reagents were of analytical grade. 4-Amino acetophenone methoxy oxime was prepared by a similar method reported earlier [1]. The ligand was prepared by refluxing a mixture of a solution containing 2-hydroxy-naphthalene-1-carbaldehyde (516.6 mg,

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Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.26 × 0.25 × 0.22 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.62 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	17636, 5967, 0.055
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4549
$N(param)_{refined}$:	446
Programs:	Bruker [13], SHELX [14]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.75995(2)	0.36359(2)	0.54960(3)	0.04258(15)
O1	0.73871(11)	0.49026(15)	0.5525(2)	0.0596(6)
O2	0.78242(9)	0.23682(14)	0.5506(2)	0.0531(5)
O3	1.02081(13)	−0.02023(18)	0.2678(3)	0.0860(9)
O4	0.47754(11)	0.76237(16)	0.7232(2)	0.0700(7)
N1	0.82561(11)	0.38926(16)	0.4706(2)	0.0413(5)
N2	0.97484(13)	0.04831(19)	0.2852(3)	0.0611(7)
N3	0.69320(10)	0.33773(16)	0.6266(2)	0.0408(5)
N4	0.51226(13)	0.68776(18)	0.6838(3)	0.0577(7)
C30	0.70183(13)	0.1618(2)	0.6308(2)	0.0418(6)
C6	0.82199(14)	0.6484(2)	0.3841(3)	0.0456(7)
C7	0.80547(13)	0.5620(2)	0.4396(2)	0.0424(6)
C21	0.67527(13)	0.0723(2)	0.6617(3)	0.0459(7)
C31	0.67484(13)	0.2525(2)	0.6505(3)	0.0440(7)
H31	0.6399	0.2502	0.6847	0.053*
C12	0.86761(12)	0.31311(19)	0.4507(2)	0.0388(6)
C11	0.83745(13)	0.4732(2)	0.4309(2)	0.0428(6)
H11	0.8706	0.4755	0.3923	0.051*
C15	0.95068(13)	0.1709(2)	0.4053(2)	0.0429(6)
C32	0.65801(12)	0.4158(2)	0.6623(2)	0.0416(6)
C29	0.75625(13)	0.1608(2)	0.5844(3)	0.0427(6)
C5	0.78723(14)	0.7348(2)	0.3925(3)	0.0488(7)
C26	0.70649(14)	−0.0156(2)	0.6489(3)	0.0468(7)
C35	0.58830(13)	0.5655(2)	0.7332(3)	0.0443(7)
C38	0.55037(14)	0.6450(2)	0.7690(3)	0.0478(7)
C27	0.76324(14)	−0.0124(2)	0.6053(3)	0.0525(8)
H27	0.7845	−0.0697	0.5980	0.063*

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Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C8	0.75690(14)	0.5642(2)	0.5009(3)	0.0472(7)
C36	0.64179(14)	0.5271(2)	0.8083(3)	0.0488(7)
H36	0.6547	0.5509	0.8843	0.059*
C18	0.99593(14)	0.0974(2)	0.3789(3)	0.0505(7)
C37	0.67683(14)	0.4537(2)	0.7728(3)	0.0480(7)
H37	0.7134	0.4300	0.8244	0.058*
C13	0.92608(14)	0.2989(2)	0.5267(3)	0.0511(7)
H13	0.9385	0.3367	0.5938	0.061*
C1	0.86896(16)	0.6530(2)	0.3190(3)	0.0566(8)
H1	0.8932	0.5984	0.3131	0.068*
C28	0.78696(15)	0.0705(2)	0.5745(3)	0.0523(8)
H28	0.8240	0.0694	0.5462	0.063*
C17	0.84954(15)	0.2543(2)	0.3538(3)	0.0582(8)
H17	0.8093	0.2622	0.3033	0.070*
C10	0.73983(15)	0.7337(2)	0.4572(3)	0.0574(8)
H10	0.7180	0.7905	0.4643	0.069*
C14	0.96667(14)	0.2276(2)	0.5030(3)	0.0543(8)
H14	1.0061	0.2182	0.5554	0.065*
C9	0.72500(16)	0.6532(2)	0.5094(3)	0.0572(8)
H9	0.6934	0.6557	0.5517	0.069*
C16	0.89014(15)	0.1843(3)	0.3306(3)	0.0594(9)
H16	0.8772	0.1454	0.2646	0.071*
C25	0.68148(16)	−0.1032(2)	0.6773(3)	0.0577(8)
H25	0.7023	−0.1605	0.6680	0.069*
C4	0.79995(17)	0.8188(2)	0.3345(3)	0.0627(9)
H4	0.7767	0.8747	0.3397	0.075*
C2	0.87998(17)	0.7364(2)	0.2636(3)	0.0657(9)
H2	0.9111	0.7369	0.2203	0.079*
C34	0.57124(16)	0.5282(3)	0.6206(3)	0.0736(11)
H34	0.5359	0.5536	0.5673	0.088*
C3	0.84538(18)	0.8203(2)	0.2710(3)	0.0676(9)
H3	0.8532	0.8764	0.2331	0.081*
C24	0.62695(19)	−0.1062(3)	0.7183(3)	0.0719(10)
H24	0.6107	−0.1649	0.7365	0.086*
C22	0.61934(16)	0.0666(2)	0.7051(3)	0.0663(9)
H22	0.5977	0.1229	0.7154	0.080*
C33	0.60539(16)	0.4542(3)	0.5860(3)	0.0735(11)
H33	0.5927	0.4300	0.5100	0.088*
C23	0.59627(18)	−0.0209(3)	0.7325(4)	0.0785(11)
H23	0.5594	−0.0226	0.7611	0.094*
C39	0.55765(19)	0.6702(3)	0.8949(3)	0.0805(12)
H39A	0.5211	0.7069	0.9036	0.121*
H39B	0.5959	0.7078	0.9217	0.121*
H39C	0.5606	0.6122	0.9405	0.121*
C19	1.06072(17)	0.0852(3)	0.4577(4)	0.0764(11)
H19A	1.0832	0.0347	0.4281	0.115*
H19B	1.0566	0.0686	0.5350	0.115*
H19C	1.0843	0.1444	0.4609	0.115*
C20	0.9945(2)	−0.0744(3)	0.1680(5)	0.1120(19)
H20A	0.9511	−0.0914	0.1678	0.168*
H20B	1.0193	−0.1321	0.1681	0.168*
H20C	0.9950	−0.0371	0.0992	0.168*
C40	0.4389(2)	0.8086(3)	0.6232(4)	0.1030(16)
H40A	0.4653	0.8291	0.5722	0.154*
H40B	0.4182	0.8636	0.6476	0.154*
H40C	0.4070	0.7642	0.5822	0.154*

3 mmol) in EtOH (5 mL) and a solution containing 1-(4-aminophenyl)ethanone *O*-methyl-oxime (492.6 mg, 3 mmol) in EtOH (5 mL) at 328 K. 7 h later, the mixture was cooled to the room temperature and filtered. The product was dried under vacuum and a yellow solid was obtained (yield 67.4%, m.p. 485–491 K). Elemental analysis: Anal. Calcd. for $C_{20}H_{18}N_2O_2$: C, 75.45%; H, 5.70%; N, 8.80%; Found: C, 75.52%; H, 5.67%; N, 8.75%.

Synthesis of the nickel(II) complex: A solution of nickel(II) acetate tetrahydrate (2.5 mg, 10 mmol) in EtOH (2 mL) was added dropwise to the solution of ligand (6.4 mg, 20 mmol) in $CHCl_3$ (4 mL) at room temperature. The color of the solution turned yellow, immediately. The mixture was filtered after being stirred for 2 h at room temperature and the filtrate was allowed to stand for 17 days at room temperature. The solvent was partially evaporated and several light yellow crystals were obtained. Anal. Calcd. for $C_{40}H_{34}N_4NiO_4$: C, 69.28%; H, 4.94%; N, 8.08%. Found: C, 69.34%; H, 4.86%; N, 8.02%.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

Oxime-type compounds are often synthesized by aldehydes or ketones with hydroxylamine or its derivatives through condensation reaction, with a general formula of $R^1R^2C=N-OR^3$ [2–4]. Oxime-type compound and their metal complexes have been widely studied and some studies on this subject are already available [5, 6]. For example: it has been used to nonlinear optical materials field and supramolecular architectures [7–12].

In the title complex, Ni1 is four-coordinated by two O atoms and two N atoms from two different ligands. The Ni1—N1 bond length is 1.894(2) Å and the Ni1—N3 bond lengths is 1.899(2) Å; and the Ni1—O1 bond length is 1.816(2) Å and the Ni1—O2 bond lengths is 1.821(2) Å, respectively. All geometric parameters are in the typical ranges.

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