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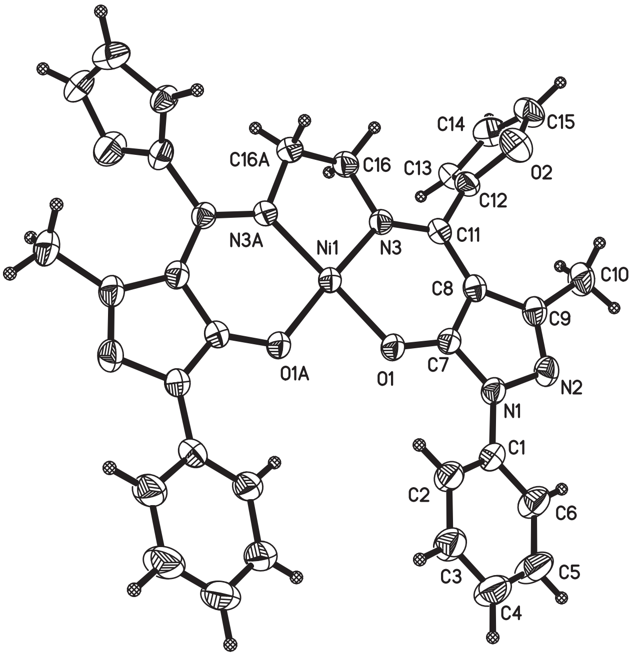
Crystal structure of (4,4′-(ethane-1,2-diylbis((nitrilo)(2-furylmethylylidene)))bis(3-methyl-1-phenyl-1*H*-pyrazol-5-olato-κ^4*N*,*N*′,*O*,*O*′))-nickel(II)), $C_{32}H_{26}N_6NiO_4$



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Abstract

$C_{32}H_{26}N_6NiO_4$, monoclinic, $C2/c$ (no. 15), $a = 22.270(4)$ Å, $b = 16.814(2)$ Å, $c = 8.1486(9)$ Å, $\beta = 111.222(3)°$, $V = 2835.0(9)$ Å^3, $Z = 4$, $R_{\mathrm{gt}}(F) = 0.0481$, $wR_{\mathrm{ref}}(F^2) = 0.1440$, $T = 295(2)$ K.

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

Table 1: Data collection and handling.

Crystal:	Green block
Size:	$0.50 \times 0.14 \times 0.12$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.73 mm^{-1}
Diffractometer, scan mode:	CCD, φ and ω
$\theta_{\max}$, completeness:	25.5°, >99%
$N(hkl)_{\mathrm{measured}}$, $N(hkl)_{\mathrm{unique}}$, R_{int}:	8993, 2647, 0.038
Criterion for I_{obs}, $N(hkl)_{\mathrm{gt}}$:	$I_{\mathrm{obs}} > 2\ \sigma(I_{\mathrm{obs}})$, 2012
$N(param)_{\mathrm{refined}}$:	197
Programs:	Bruker [1], SHELX [2], ORTEP [3]

Source of material

A mixture of 40 mL double Schiff base ligand (4.2 g, 7.5 mmol) dimethyl sulfoxide solution and 35 mL $Ni(CH_3COO)_2·H_2O$ (1.5 g, 7.5 mmol) ethanol solution was refluxed for 2 h at 353 K. A green product which precipitated was filtered off, washed several times with anhydrous ethanol, and dried in air, yield: 71%. The green powder was recrystallized from dimethyl formamide and crystals were obtained at room temperature after several days.

Experimental details

There is a minimal O/CH disorder in the furyl moiety (O3/C13), which was not worth to be considered. Consequently, the highest difference electron density peak is near C13.

Comment

Acylpyrazolones and its Schiff base compounds have spectroscopic properties, antibacterial and antiviral activity [5]. The Schiff bases derived from the reaction of acylpyrazolones with amines are an excellence class of ligands which have a variety of applications including biological, clinical, industrial, analytical, as well as catalysis and organic synthesis [6–9]. In our previous publications we studied the 4-heterocyclic acylpyrazolone Schiff base complexes, which exhibit certain electron transfer effect, ferromagnetism and electrostatic interactions with DNA [10, 11]. 1-Phenyl-3-methyl-4-(2-furoyl)-5-pyrazolone (HPMFP), is a member of a family of 4-heterocyclic acylpyrazolones, first synthesized in 1983 [12]. In recent years, we have reported on double Schiff bases derived from HPMFP and their complexes, which possess high electrocatalytic activity [4].

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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}
C1	0.89166(15)	0.2585(3)	0.8847(3)	0.0516(8)
C2	0.94733(18)	0.2972(3)	0.8746(4)	0.0631(10)
H2	0.980110	0.256286	0.870950	0.076*
C3	0.9532(2)	0.3977(3)	0.8701(5)	0.0771(12)
H3	0.990627	0.424157	0.864213	0.093*
C4	0.9057(2)	0.4595(3)	0.8740(5)	0.0856(13)
H4	0.910317	0.527013	0.870357	0.103*
C5	0.8507(2)	0.4194(3)	0.8835(6)	0.0933(15)
H5	0.817717	0.460454	0.885266	0.112*
C6	0.84376(19)	0.3195(3)	0.8904(5)	0.0748(12)
H6	0.806719	0.293483	0.899013	0.090*
C7	0.91626(14)	0.0804(2)	0.8594(3)	0.0453(7)
C8	0.88991(15)	−0.0073(2)	0.8881(4)	0.0463(8)
C9	0.84059(16)	0.0242(3)	0.9390(4)	0.0537(9)
C10	0.79511(18)	−0.0346(3)	0.9855(5)	0.0730(11)
H10A	0.819067	−0.073194	1.067796	0.110*
H10B	0.770398	−0.077089	0.908910	0.110*
H10C	0.766631	0.008656	1.009832	0.110*
C11	0.90910(14)	−0.0997(2)	0.8549(3)	0.0441(7)
C12	0.87420(15)	−0.1870(3)	0.8760(4)	0.0518(7)
C13	0.82907(14)	−0.2361(2)	0.7755(4)	0.0520(8)
H13	0.814590	−0.224384	0.677315	0.062*
C14	0.80728(18)	−0.3086(3)	0.8443(5)	0.0707(11)
H14	0.775129	−0.353916	0.799368	0.085*
C15	0.83928(19)	−0.3028(3)	0.9826(5)	0.0724(11)
H15	0.834073	−0.343068	1.052540	0.087*
C16	0.96697(15)	−0.2074(2)	0.7551(4)	0.0532(8)
H16A	0.934595	−0.223877	0.662848	0.064*
H16B	0.965190	−0.255831	0.824556	0.064*
N1	0.88420(12)	0.1557(2)	0.8929(3)	0.0498(7)
N2	0.83674(13)	0.1202(2)	0.9425(3)	0.0579(8)
N3	0.95431(11)	−0.10914(18)	0.7997(3)	0.0445(6)
Ni1	1.000000	−0.00977(4)	0.750000	0.0436(2)
O1	0.96051(10)	0.09334(16)	0.8066(2)	0.0514(6)
O2	0.88314(13)	−0.2242(2)	1.0060(3)	0.0803(8)

The structure of the title compound is illustrated in the figure. The coordination around Ni(II) is square planar, coordinated by two oxygen atoms and two imido group nitrogen atoms from tetradentate Schiff base ligand. The Ni is located in the coordination center forming a stable chelate ring and complex arranged rules along the c axis. The Ni(1)—O(1), Ni(1)—N(3) bond length are 1.854(2) Å and 1.868(3) Å. The O(1)—Ni(1)—N(3) angle is 96.04(11)°, while N(3)—Ni(1)—N(3A), O(1)—Ni(1)—O(1A), O(1)—Ni(1)—N(3A) angles are 86.88(11)°, 81.21(13)° and 175.12(9)°, respectively. The bond length of Schiff base ligand is different from the title complex. In the complex, the C(7)—O(1) bond length is 1.287(4) Å. The bond lengths of C(7)—C(8) and C(11)—C(8) are 1.407(4) Å and 1.409(4) Å. Moreover, the C(11)—N(3) bond length is 1.318(3) Å, which is shorter than 1.330 Å of C(11)—N(3) bond length of the free ligand [13]. These parameters indicate that the oxygens and nitrogens of the ligand participate in coordination. In the complex, the bond lengths of O(2)—C(12) and O(2)—C(15) at the furan ring are 1.338(4) Å and 1.405(4) Å, respectively.

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