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Crystal structure of (4,4′-(ethane-1,2-diylbis((nitrilo)(2-furylmethylene))bis(3-methyl-1-phenyl-1H-pyrazol-5-olato-κ²N,N′,O,O′))-nickel(II)), C₃₂H₂₆N₆NiO₄

Abstract

C₃₂H₂₆N₆NiO₄, monoclinic, C2/c (no. 15), a = 22.270(4) Å, b = 16.814(2) Å, c = 8.1486(9) Å, β = 111.222(3)°, V = 2835.0(9) Å³, Z = 4, R₁(F) = 0.0481, wR₂(F²) = 0.1440, T = 295(2) K.

Crystal: Green block
Size: 0.50 × 0.14 × 0.12 mm
Wavelength: Mo Kα radiation (0.71073 Å)
µ: 0.73 mm⁻¹
Diffraactometer, scan mode: CCD, φ and ω
θmax, completeness: 25.5°, >99%
N(hkl)measured, N(hkl)unique, Rint: 8993, 2647, 0.038
Criterion for I(obs), N(hkl)gt: I(obs) > 2σ(I(obs)), 2012
N(param)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(CH₃COO)₂·H₂O (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].
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 groups of nitrogen atoms from tetradentate Schiff base ligand. The bond length of Schiff base ligand is different from 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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References