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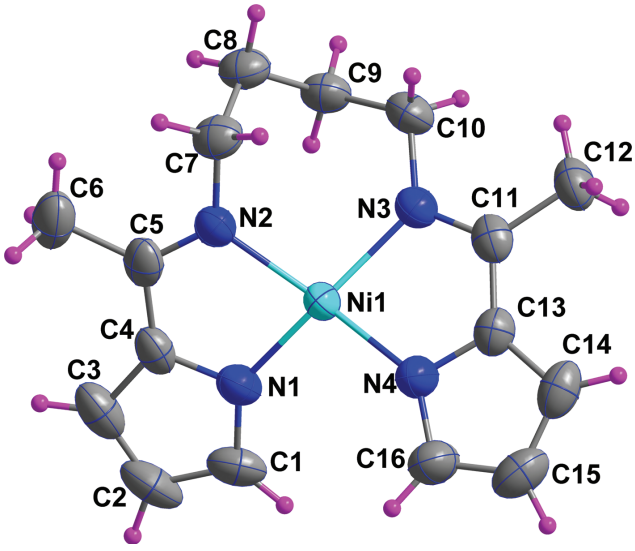
Crystal structure of [*N*,*N*-bis((pyrrol-2-yl) ethylidene)butane-1,4-diamine-κ^4*N*,*N*′,*N*″,*N*‴]-nickel(II), $C_{16}H_{20}N_4Ni$



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Abstract

$C_{16}H_{20}N_4Ni$, monoclinic, $P2_1/n$ (no. 14), $a = 7.6578(10)$ Å, $b = 17.184(2)$ Å, $c = 11.6581(15)$ Å, $\beta = 100.837(2)°$, $V = 1506.7(3)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0356$, $wR_{ref}(F^2) = 0.0945$, $T = 296(2)$ 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.

Source of material

2-Acetylpyrrole (0.218 g, 2 mmol) and butane-1,4-diamine (0.088 g, 1 mmol) were dissolved in an ethanol/THF (1:1)

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Table 1: Data collection and handling.

Crystal:	Block, brown
Size:	0.15 × 0.14 × 0.12 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.29 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
$2\theta_{max}$, completeness:	26°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8560, 2962, 0.036
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2169
$N(param)_{refined}$:	190
Programs:	Bruker programs [1], SHELXT [2], SHELXL [3]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.35238(4)	0.225436(19)	0.37149(3)	0.03754(14)
N1	0.4070(3)	0.16387(14)	0.2449(2)	0.0479(6)
N2	0.3022(3)	0.30600(13)	0.24925(19)	0.0444(6)
N3	0.3919(3)	0.29280(13)	0.5143(2)	0.0430(6)
N4	0.3022(3)	0.14746(13)	0.4783(2)	0.0502(6)
C1	0.4674(4)	0.09253(18)	0.2236(3)	0.0584(8)
H1A	0.4883	0.0521	0.2774	0.070*
C2	0.4936(4)	0.0888(2)	0.1090(3)	0.0681(10)
H2A	0.5349	0.0461	0.0730	0.082*
C3	0.4469(4)	0.1605(2)	0.0583(3)	0.0631(9)
H3A	0.4502	0.1752	−0.0180	0.076*
C4	0.3944(4)	0.20564(18)	0.1436(2)	0.0482(7)
C5	0.3324(4)	0.28466(18)	0.1482(2)	0.0481(7)
C6	0.3062(4)	0.3345(2)	0.0407(2)	0.0680(10)
H6A	0.2651	0.3851	0.0586	0.102*
H6B	0.2197	0.3108	−0.0194	0.102*
H6C	0.4170	0.3396	0.0142	0.102*
C7	0.2404(4)	0.38381(16)	0.2725(3)	0.0532(8)
H7A	0.1599	0.3801	0.3273	0.064*
H7B	0.1751	0.4059	0.2005	0.064*
C8	0.3929(4)	0.43676(17)	0.3219(3)	0.0546(8)
H8A	0.3452	0.4841	0.3492	0.066*
H8B	0.4549	0.4510	0.2596	0.066*
C9	0.5262(4)	0.40214(17)	0.4216(2)	0.0504(7)
H9A	0.5843	0.3587	0.3911	0.061*
H9B	0.6167	0.4410	0.4479	0.061*
C10	0.4553(4)	0.37398(17)	0.5264(3)	0.0529(8)
H10A	0.5483	0.3781	0.5952	0.063*
H10B	0.3579	0.4074	0.5379	0.063*

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C11	0.3628(4)	0.25557(18)	0.6061(2)	0.0462(7)
C12	0.3799(4)	0.29090(19)	0.7251(2)	0.0599(9)
H12A	0.4149	0.3444	0.7224	0.090*
H12B	0.4680	0.2630	0.7791	0.090*
H12C	0.2676	0.2879	0.7502	0.090*
C13	0.3122(4)	0.17546(17)	0.5887(2)	0.0451(7)
C14	0.2711(4)	0.1170(2)	0.6607(3)	0.0599(9)
H14A	0.2691	0.1210	0.7400	0.072*
C15	0.2335(4)	0.0510(2)	0.5917(3)	0.0670(10)
H15A	0.2007	0.0024	0.6154	0.080*
C16	0.2544(4)	0.07186(18)	0.4812(3)	0.0618(9)
H16A	0.2378	0.0385	0.4172	0.074*

solution (10 mL). The mixture was stirred for 4 h at room temperature, and then nickel acetate (0.175 g, 1 mmol) was added. The resulting solution was left in air for a few days, yielding brown block-shaped crystals.

Experimental details

The hydrogen atoms were placed at calculated positions and refined as riding atoms with isotropic displacement parameters.

Discussion

Schiff base ligands bearing pyrrole units have attracted much recent attention due to their excellent coordination abilities for metal ions [4, 5]. The crystal structures of copper(II) and zinc(II) complexes with bis(pyrrol-2-ylmethyleneamine) ligands have been weidely investigated. By varying the spacers between two pyrrol-2-yl-methyleneamine units, dinuclear dimeric helicates, trinuclear trimeric triangles, and tetranuclear tetrameric square complexes could be generated [4–6]. As part of our ongoing studies of pyrrol-2-ylmethyleneamine-metals [6–8], the title complex was synthesized and characterized by X-ray diffraction. In the title complex, the Ni(II) ion is surrounded by the ligand *via* its four N atoms, thus giving a distorted square planar geometry (r.m.s. deviation from planarity 0.2947 Å). The dihedral angle, defined by the intersection of two pyrrol-2-ylmethyleneamine planes at the nickel center, is 34.5(2)°. The N—Ni—N angles range from 82.68(10) to 159.09(9)°, and the bond distances of Ni—N span from 1.917(2) to 2.003(2) Å. As expected, there exist no classical hydrogen bonds in the crystal.

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