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Crystal structure of [5,5'-((propane-1,3-diylbis(azanylylidene))bis(ethan-1-yl-2-ylidene))bis(3-(ethoxycarbonyl)-2,4-dimethylpyrrol-1-ido)- κ^4N,N',N'',N''']nickel(II), $C_{23}H_{30}N_4O_4Ni$

Table 1: Data collection and handling.

Crystal:	Brown block
Size:	0.19 × 0.18 × 0.16 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.89 mm ⁻¹
Diffractometer, scan mode:	SMART detector, φ and ω
$\theta_{\max}$, completeness:	25.0°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	5995, 3976, 0.053
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3471
$N(\text{param})_{\text{refined}}$:	293
Programs:	SHELX [1], Bruker [2]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	0.47371(4)	0.47536(2)	0.23179(2)	0.03240(15)
O1	1.0777(3)	0.84442(17)	0.46845(17)	0.0530(5)
O2	1.1071(4)	0.7695(2)	0.62107(19)	0.0755(8)
O3	0.2304(4)	0.85102(18)	-0.03955(18)	0.0626(6)
O4	0.2230(5)	0.8981(2)	0.1320(2)	0.0875(9)
N1	0.6577(3)	0.56542(17)	0.34353(17)	0.0339(5)
N2	0.5053(3)	0.35975(18)	0.31607(19)	0.0412(5)
N3	0.3404(3)	0.37672(17)	0.10850(18)	0.0386(5)
N4	0.3805(3)	0.58554(17)	0.15816(17)	0.0338(5)
C1	1.2515(5)	1.0168(3)	0.4415(4)	0.0748(11)
H1A	1.346984	1.080302	0.474448	0.112*
H1B	1.286678	0.979963	0.381153	0.112*
H1C	1.140436	1.041261	0.417786	0.112*
C2	1.2213(5)	0.9386(3)	0.5200(3)	0.0698(10)
H2A	1.333044	0.913705	0.544708	0.084*
H2B	1.186261	0.975229	0.581477	0.084*
C3	1.0304(4)	0.7633(2)	0.5288(2)	0.0436(7)
C4	0.8824(3)	0.6730(2)	0.4719(2)	0.0364(6)
C5	0.7834(3)	0.6625(2)	0.3646(2)	0.0351(6)
C7	0.6824(3)	0.5112(2)	0.4340(2)	0.0346(6)
C6	0.8159(4)	0.5745(2)	0.5150(2)	0.0362(6)
C8	0.8126(4)	0.7365(2)	0.2809(2)	0.0475(7)
H8A	0.928851	0.734779	0.261483	0.071*
H8B	0.811018	0.811622	0.308357	0.071*
H8C	0.716646	0.710687	0.218737	0.071*
C9	0.8813(4)	0.5423(3)	0.6228(2)	0.0452(7)
H9A	1.011831	0.550167	0.635001	0.068*
H9B	0.823745	0.466068	0.625006	0.068*
H9C	0.850105	0.590170	0.677535	0.068*
C10	0.5941(4)	0.3959(2)	0.4129(2)	0.0391(6)
H10	0.600086	0.349205	0.465823	0.047*
C11	0.4328(5)	0.2388(2)	0.2863(3)	0.0672(10)
H11A	0.532287	0.204076	0.272526	0.081*

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Abstract

$C_{23}H_{30}N_4O_4Ni$, triclinic, $P\bar{1}$ (no. 2), $a = 7.5883(9)$ Å, $b = 12.3110(15)$ Å, $c = 12.7718(15)$ Å, $\alpha = 95.621(2)^\circ$, $\beta = 99.908(2)^\circ$, $\gamma = 101.30(2)^\circ$, $V = 1141.8(2)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0433$, $wR_{\text{ref}}(F^2) = 0.1239$, $T = 296$ K.

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The crystal structure is shown in the figure (Hydrogen atoms are omitted for clarity). Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

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Table 2 (continued)

Atom	x	y	z	U _{iso} */U _{eq}
H11B	0.389533	0.208710	0.346951	0.081*
C12	0.2832(6)	0.2070(3)	0.1923(3)	0.0777(12)
H12A	0.176006	0.229212	0.211640	0.093*
H12B	0.253979	0.126162	0.176051	0.093*
C13	0.3153(5)	0.2537(2)	0.0931(3)	0.0548(8)
H13A	0.423588	0.233811	0.073035	0.066*
H13B	0.211730	0.221659	0.035292	0.066*
C14	0.2855(4)	0.4266(2)	0.0287(2)	0.0393(6)
H14	0.235825	0.387603	−0.039456	0.047*
C15	0.3052(3)	0.5443(2)	0.0506(2)	0.0355(6)
C16	0.2426(4)	0.6241(2)	−0.0036(2)	0.0377(6)
C17	0.2721(4)	0.7187(2)	0.0746(2)	0.0395(6)
C18	0.3526(4)	0.6898(2)	0.1735(2)	0.0364(6)
C19	0.1509(4)	0.6069(3)	−0.1205(2)	0.0490(7)
H19A	0.230998	0.648133	−0.160356	0.074*
H19B	0.038729	0.632932	−0.127572	0.074*
H19C	0.124808	0.528837	−0.147906	0.074*
C20	0.3918(4)	0.7577(2)	0.2821(2)	0.0488(7)
H20A	0.279493	0.771931	0.299411	0.073*
H20B	0.474493	0.827480	0.281679	0.073*
H20C	0.446539	0.717357	0.334676	0.073*
C21	0.2384(4)	0.8297(3)	0.0615(3)	0.0493(7)
C22	0.2068(7)	0.9618(3)	−0.0599(3)	0.0792(12)
H22A	0.080746	0.967060	−0.060461	0.095*
H22B	0.285429	1.017648	−0.003522	0.095*
C23	0.2532(9)	0.9817(4)	−0.1620(4)	0.120(2)
H23A	0.237913	1.054885	−0.175972	0.180*
H23B	0.378323	0.977021	−0.160669	0.180*
H23C	0.174262	0.926588	−0.217436	0.180*

Source of material

Ethyl 5-formyl-2,4-dimethyl-1*H*-pyrrole-3-carboxylate (0.390 g, 2 mmol) and propane-1,3-diamine (0.074 g, 1 mmol) were dissolved in an ethanol/THF (1:1) 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 crystals.

Experimental details

The structure was solved by direct methods and refined with the SHELX software package [7]. The hydrogen atoms were placed at calculated positions and refined as riding atoms.

Comment

Schiff base ligands bearing pyrrole units have attracted much recent attention due to their excellent coordination

abilities [3, 4]. The crystal structures of copper(II) and zinc(II) complexes with bis(pyrrol-2-ylmethylethylamine) ligands have been investigated [3–8], while those of the nickel(II) complexes are rarely reported [7].

In the title complex, the Ni(II) ion is coordinated by the ligand *via* its four N atoms, giving a distorted square planar geometry (r.m.s. deviation 0.2330 Å). The dihedral angle, defined by the intersection of two pyrrol-2-ylmethylethylamine planes (N1/C4–C7, r.m.s. deviation 0.0115 Å; N4/C15–C18, r.m.s. deviation 0.0159 Å) at the nickel center, is 45.4°. The N–Cu–N angles range from 83.70(9) to 166.03(10)°, and the bond distances of Ni–N span from 1.890(2) to 1.903(2) Å. The molecular structure of the title complex is isostructural with that of a copper(II) complex [8]. Similarly, there exist no classical hydrogen bonds in the crystal.

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