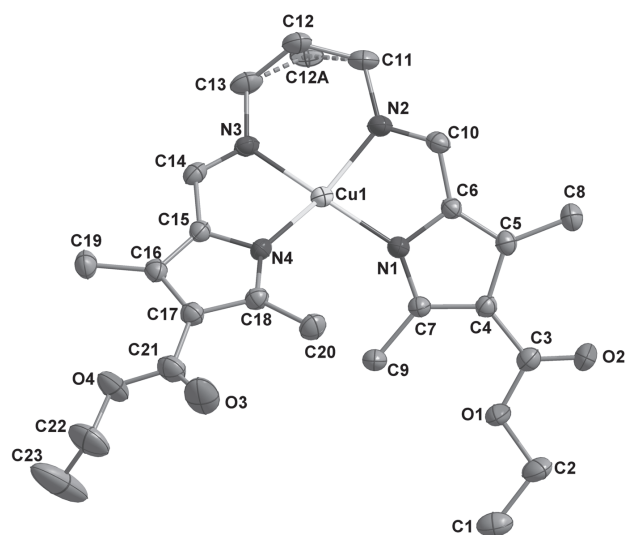


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Crystal structure of {5,5'-((propane-1,3-diylbis(azanylylidene))bis(methanylylidene))bis(3-(ethoxycarbonyl)-2,4-dimethylpyrrol-1-ido)- κ^4N,N',N'',N''' }zinc(II), $C_{23}H_{30}N_4O_4Cu$



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Abstract

$C_{13}H_{17}N_3OS$, monoclinic, $P\bar{1}$ (no. 2), $a = 7.746(6)$ Å, $b = 12.344(9)$ Å, $c = 12.655(9)$ Å, $\alpha = 95.766(7)^\circ$, $\beta = 98.007(7)^\circ$, $\gamma = 101.595(13)^\circ$, $V = 1163.4(15)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0550$, $wR_{ref}(F^2) = 0.1616$, $T = 296(2)$ 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 1: Data collection and handling.

Crystal:	Brown block
Size:	$0.20 \times 0.16 \times 0.15$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.98 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω -scans
$\theta_{\max}$, completeness:	27.5° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	12740, 5310, 0.066
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4099
$N(\text{param})_{\text{refined}}$:	305
Programs:	Bruker programs [1], SHELX [2]

Source of material

Ethyl 5-formyl-2,4-dimethyl-1H-pyrrole-3-carboxylate (2 mmol) and propane-1,3-diamine (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 copper acetate monohydrate (1 mmol) was added. The resulting solution was left in air for a few days, yielding brown block-shaped crystals.

Experimental details

The C12 atom splitted over two positions, with the occupancy value of C12/C12A being 0.72/0.28. 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 attention due to their excellent coordination abilities [3, 4]. The crystal structures of copper(II) and zinc(II) complexes with bis(pyrrol-2-ylmethyleneamine) ligands have been widely investigated [3–6]. As part of our ongoing studies of pyrrol complexes [5–7], the title complex was synthesized and characterized.

In the title complex, the Cu(II) ion is surrounded by the ligand *via* its four N atoms, thus giving a distorted square planar geometry (r.m.s. deviation 0.2679 Å). The dihedral angle, defined by the intersection of two pyrrol-2-ylmethyleneamine planes (N1/C4–C7, r.m.s. deviation 0.0067 Å; N4/C15–C18, r.m.s. deviation 0.0091 Å) at the copper center, is 43.3° . The N–Cu–N angles range from $82.94(13)$ to $160.35(12)^\circ$, and the bond distances of Cu–N span from 1.965(3) to 1.977(3) Å.

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
Cu1	0.52283(5)	0.51864(3)	0.26879(3)	0.03900(16)
O1	−0.0819(4)	0.1584(2)	0.0234(2)	0.0561(7)
O2	−0.0996(5)	0.2388(3)	−0.1273(3)	0.0801(10)
O3	0.7828(7)	0.0999(3)	0.3772(3)	0.1045(14)
O4	0.7967(5)	0.1575(3)	0.5516(3)	0.0808(10)
N1	0.3297(4)	0.4329(2)	0.1560(2)	0.0398(6)
N2	0.5055(4)	0.6401(2)	0.1799(3)	0.0480(7)
N3	0.6472(4)	0.6234(2)	0.3979(2)	0.0437(7)
N4	0.6320(4)	0.4110(2)	0.3439(2)	0.0398(6)
C1	−0.2559(7)	−0.0151(4)	0.0457(4)	0.0745(13)
H1B	−0.346541	−0.078398	0.010909	0.112 [*]
H1C	−0.295498	0.019143	0.106708	0.112 [*]
H1A	−0.148002	−0.039018	0.069256	0.112 [*]
C2	−0.2213(6)	0.0666(4)	−0.0311(4)	0.0703(12)
H2A	−0.184334	0.032270	−0.094075	0.084 [*]
H2B	−0.328770	0.092661	−0.054224	0.084 [*]
C3	−0.0289(5)	0.2417(3)	−0.0355(3)	0.0476(8)
C4	0.1158(4)	0.3298(3)	0.0242(3)	0.0390(7)
C5	0.1845(4)	0.4302(3)	−0.0156(3)	0.0407(7)
C6	0.3126(4)	0.4915(3)	0.0675(3)	0.0375(7)
C7	0.2081(4)	0.3355(3)	0.1305(3)	0.0381(7)
C8	0.1285(5)	0.4666(3)	−0.1220(3)	0.0493(8)
H8A	0.155598	0.417937	−0.178534	0.074 [*]
H8B	0.191704	0.541634	−0.123305	0.074 [*]
H8C	0.002358	0.463403	−0.132527	0.074 [*]
C9	0.1787(6)	0.2566(3)	0.2115(3)	0.0525(9)
H9A	0.269417	0.281107	0.273996	0.079 [*]
H9B	0.184540	0.183255	0.180928	0.079 [*]
H9C	0.063336	0.254550	0.231828	0.079 [*]
C10	0.4089(5)	0.6052(3)	0.0865(3)	0.0444(8)
H10	0.402254	0.652080	0.033722	0.053 [*]
C11	0.5866(7)	0.7590(3)	0.2090(3)	0.0729(13)
H11A ^a	0.491730	0.798797	0.215127	0.087 [*]
H11B ^a	0.645486	0.784742	0.150467	0.087 [*]
H11C ^b	0.522523	0.801686	0.163575	0.087 [*]
H11D ^b	0.709387	0.772895	0.196546	0.087 [*]
C12 ^a	0.7173(8)	0.7896(4)	0.3096(4)	0.063(2)
H12A ^a	0.746445	0.870375	0.324823	0.076 [*]
H12B ^a	0.825535	0.766775	0.295590	0.076 [*]
C12A ^b	0.5823(18)	0.7960(9)	0.3232(5)	0.055(5)
H12C ^b	0.633526	0.875524	0.336605	0.066 [*]
H12D ^b	0.457603	0.786575	0.331264	0.066 [*]
C13	0.6702(6)	0.7450(3)	0.4103(3)	0.0617(11)
H13A ^a	0.764287	0.778241	0.470452	0.074 [*]
H13B ^a	0.560500	0.764841	0.425765	0.074 [*]
H13C ^b	0.797300	0.777206	0.420707	0.074 [*]
H13D ^b	0.628002	0.767291	0.476085	0.074 [*]
C14	0.7157(5)	0.5735(3)	0.4733(3)	0.0437(8)
H14	0.769580	0.612310	0.540455	0.052 [*]
C15	0.7055(4)	0.4567(3)	0.4495(3)	0.0396(7)
C16	0.7705(5)	0.3801(3)	0.5073(3)	0.0433(8)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C17	0.7412(5)	0.2822(3)	0.4313(3)	0.0455(8)
C18	0.6567(5)	0.3058(3)	0.3317(3)	0.0418(7)
C19	0.8600(6)	0.4010(4)	0.6228(3)	0.0566(10)
H19A	0.976269	0.384026	0.627497	0.085 [*]
H19B	0.789333	0.354458	0.664364	0.085 [*]
H19C	0.871817	0.477952	0.650375	0.085 [*]
C20	0.6122(6)	0.2342(3)	0.2252(3)	0.0580(10)
H20A	0.541423	0.267680	0.174982	0.087 [*]
H20B	0.546081	0.161697	0.233051	0.087 [*]
H20C	0.720488	0.227201	0.199003	0.087 [*]
C21	0.7753(6)	0.1715(4)	0.4471(4)	0.0628(11)
C22	0.8274(9)	0.0514(4)	0.5749(6)	0.111(2)
H22A	0.952239	0.058600	0.604320	0.134 [*]
H22B	0.799019	−0.000951	0.509240	0.134 [*]
C23	0.7161(16)	0.0102(6)	0.6521(8)	0.189(5)
H23A	0.592478	−0.000566	0.621179	0.283 [*]
H23B	0.741720	0.063499	0.716022	0.283 [*]
H23C	0.740590	−0.059486	0.670179	0.283 [*]

Occupancies: ^a = 0.721(11), ^b = 0.279(11).

The molecular structure of the title complex is similar to that of [N,N-bis((pyrrol-2-yl)methylidene)butane-1,4-diamine-κ⁴N,N',N'',N''']copper(II) [1].

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