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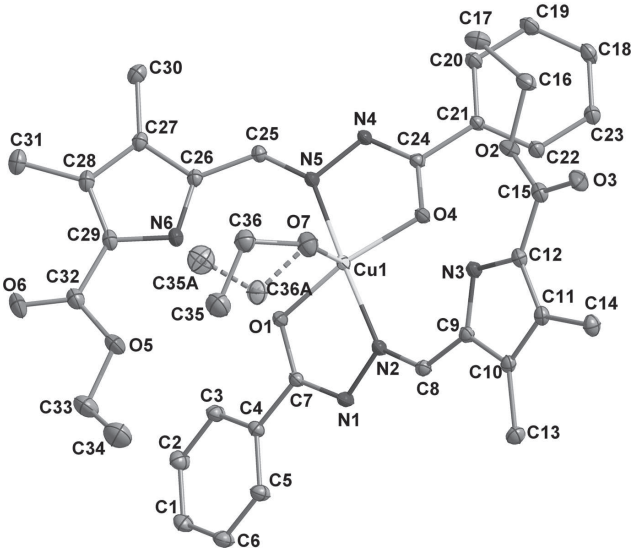
Crystal structure of ethanol-bis(*N*-((5-(ethoxycarbonyl)-3,4-dimethyl-1*H*-pyrrol-2-yl)methylene)benzohydrazonato-κ^2N,O)copper(II), $C_{36}H_{42}N_6O_7Cu$



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Abstract

$C_{20}H_29N_3O_5$, triclinic, $P\bar{1}$ (no. 2), $a = 8.482(6)$ Å, $b = 15.272(12)$ Å, $c = 15.285(12)$ Å, $a = 112.024(11)°$, $\beta = 95.325(9)°$, $\gamma = 98.958(9)°$, $V = 1788(2)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0570$, $wR_{ref}(F^2) = 0.1687$, $T = 296(2)$ K.

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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

The title complex was prepared according to the literature method while using ethanol as solvent [5].

Experimental details

The structure was solved by direct methods and refined with the SHELX software package [1]. Each of the carbon atoms of

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Table 1: Data collection and handling.

Crystal:	Brown block
Size:	0.16 × 0.14 × 0.13 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.67 mm^{-1}
Diffractometer, scan mode:	SMART detector, φ and ω
θ_{max}, completeness:	27.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	18896, 8026, 0.055
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5977
$N(param)_{refined}$:	473
Programs:	SHELX [1], Bruker [2]

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

Atom	x	y	z	U_{iso}*/U_{eq}
Cu1	0.58033(4)	0.65052(2)	0.22980(2)	0.04688(14)
O1	0.5943(3)	0.52211(14)	0.21912(15)	0.0547(5)
O2	0.5433(3)	1.00774(17)	0.33665(18)	0.0663(6)
O3	0.6248(4)	1.13971(17)	0.4739(2)	0.0846(8)
O4	0.6426(3)	0.77094(14)	0.21670(14)	0.0559(5)
O5	0.3041(4)	0.3346(2)	0.2342(2)	0.0926(9)
O6	0.1055(4)	0.2098(2)	0.1409(3)	0.0980(10)
O7	0.3729(4)	0.6933(2)	0.3228(2)	0.0935(9)
H7	0.374025	0.750173	0.362237	0.140*
N1	0.7809(3)	0.60301(17)	0.35735(18)	0.0527(6)
N2	0.7329(3)	0.68563(17)	0.35240(17)	0.0479(5)
N3	0.6896(3)	0.89660(16)	0.40507(17)	0.0490(6)
H3	0.637085	0.865698	0.347808	0.059*
N4	0.4882(3)	0.68792(16)	0.06650(17)	0.0474(6)
N5	0.4470(3)	0.61425(16)	0.10065(17)	0.0449(5)
N6	0.2918(3)	0.42428(17)	0.11428(19)	0.0523(6)
H6	0.366734	0.452728	0.163565	0.063*
C1	0.8189(5)	0.2608(3)	0.2808(3)	0.0761(11)
H1	0.845892	0.203568	0.279357	0.091*
C2	0.7431(5)	0.2645(3)	0.2000(3)	0.0787(11)
H2	0.717326	0.209367	0.143506	0.094*
C3	0.7039(5)	0.3500(2)	0.2015(3)	0.0667(9)
H3A	0.652517	0.351906	0.145979	0.080*
C4	0.7408(4)	0.4323(2)	0.2851(2)	0.0502(7)
C5	0.8189(5)	0.4275(3)	0.3665(3)	0.0674(9)
H5	0.846894	0.482567	0.423072	0.081*
C6	0.8553(5)	0.3424(3)	0.3645(3)	0.0799(11)
H6A	0.904852	0.339734	0.420115	0.096*
C7	0.7027(4)	0.5248(2)	0.2873(2)	0.0472(6)
C8	0.7967(4)	0.7616(2)	0.4281(2)	0.0501(7)
H8	0.867249	0.751038	0.471685	0.060*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C9	0.7778(4)	0.8589(2)	0.4566(2)	0.0472(6)
C10	0.8446(4)	0.9323(2)	0.5453(2)	0.0492(7)
C11	0.7961(4)	1.0165(2)	0.5478(2)	0.0515(7)
C12	0.6999(4)	0.9928(2)	0.4611(2)	0.0506(7)
C13	0.9503(4)	0.9218(3)	0.6240(2)	0.0657(9)
H13A	0.980811	0.982700	0.677806	0.099*
H13B	0.891763	0.874586	0.642934	0.099*
H13C	1.045655	0.901280	0.601316	0.099*
C14	0.8405(5)	1.1143(2)	0.6300(3)	0.0683(9)
H14A	0.749994	1.125930	0.662875	0.102*
H14B	0.868765	1.163276	0.605828	0.102*
H14C	0.931108	1.115744	0.673498	0.102*
C15	0.6210(4)	1.0553(2)	0.4263(3)	0.0599(8)
C16	0.4595(5)	1.0633(3)	0.2954(3)	0.0842(12)
H16A	0.537267	1.107710	0.281099	0.101*
H16B	0.397049	1.100343	0.340201	0.101*
C17	0.3513(6)	0.9946(4)	0.2067(3)	0.0942(14)
H17A	0.294701	1.029463	0.178078	0.141*
H17B	0.414359	0.958446	0.162722	0.141*
H17C	0.274698	0.951109	0.221589	0.141*
C18	0.7673(5)	0.9906(2)	0.0459(3)	0.0730(10)
H18	0.807865	1.039325	0.026520	0.088*
C19	0.6362(6)	0.9190(3)	−0.0093(3)	0.0821(12)
H19	0.586641	0.920526	−0.065423	0.099*
C20	0.5776(5)	0.8451(2)	0.0178(2)	0.0651(9)
H20	0.490383	0.796543	−0.020706	0.078*
C21	0.6484(4)	0.84323(19)	0.10220(19)	0.0447(6)
C22	0.7765(4)	0.9159(2)	0.1578(2)	0.0626(9)
H22	0.823670	0.916295	0.215375	0.075*
C23	0.8367(5)	0.9892(2)	0.1290(3)	0.0731(10)
H23	0.924807	1.037438	0.166884	0.088*
C24	0.5881(4)	0.76268(19)	0.13058(19)	0.0443(6)
C25	0.3392(3)	0.5432(2)	0.0385(2)	0.0475(6)
H25	0.307667	0.550182	−0.018032	0.057*
C26	0.2611(4)	0.4555(2)	0.0433(2)	0.0492(7)
C27	0.1285(4)	0.3899(2)	−0.0230(2)	0.0536(7)
C28	0.0796(4)	0.3176(2)	0.0105(3)	0.0594(8)
C29	0.1836(4)	0.3399(2)	0.0943(3)	0.0583(8)
C30	0.0529(4)	0.3934(3)	−0.1139(3)	0.0714(10)
H30A	0.107805	0.449808	−0.120790	0.107*
H30B	0.061523	0.336742	−0.167315	0.107*
H30C	−0.059186	0.395945	−0.111926	0.107*
C31	−0.0602(5)	0.2326(3)	−0.0370(3)	0.0863(13)
H31A	−0.111683	0.235079	−0.094450	0.130*
H31B	−0.021106	0.173791	−0.052588	0.130*
H31C	−0.136922	0.234954	0.005817	0.130*
C32	0.1903(5)	0.2871(3)	0.1566(3)	0.0718(10)
C33	0.3283(9)	0.2863(4)	0.2984(4)	0.132(2)
H33A	0.232411	0.237764	0.288707	0.158*
H33B	0.418701	0.254288	0.284742	0.158*
C34	0.3607(10)	0.3564(5)	0.3972(4)	0.158(3)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H34A	0.376629	0.324069	0.439410	0.237*
H34B	0.456339	0.403962	0.406750	0.237*
H34C	0.270486	0.387477	0.410706	0.237*
C35[a]	0.268(2)	0.5817(11)	0.3880(10)	0.133(5)
H35A[a]	0.372310	0.565734	0.383313	0.199*
H35B[a]	0.186499	0.523685	0.368603	0.199*
H35C[a]	0.264697	0.622770	0.452957	0.199*
C35A[b]	0.169(3)	0.594(2)	0.357(2)	0.191(13)
H35D[b]	0.119021	0.638168	0.338699	0.287*
H35E[b]	0.136542	0.593114	0.415010	0.287*
H35F[b]	0.135750	0.530250	0.306922	0.287*
C36[a]	0.2350(11)	0.6329(6)	0.3247(7)	0.073(3)
H36A[a]	0.191864	0.585755	0.260299	0.088*
H36B[a]	0.153867	0.670573	0.347006	0.088*
C36A[b]	0.326(2)	0.6208(12)	0.3696(14)	0.093(6)
H36C[b]	0.373179	0.564798	0.341509	0.112*
H36D[b]	0.368146	0.650902	0.437544	0.112*

[a]Occupancy: 0.61(2), [b]Occupancy: 0.39(2).

ethanol molecule occupied two positions with the occupancy ratio of C35(C36)/C35A(C36A) being 0.61/0.39. All hydrogen atoms were placed at calculated positions.

Comment

Acylhydrazones are an important class of ligands in coordination chemistry and have been found extensive application [3]. Our previous work shows that acylhydrazone ligands bearing pyrrole units and their complexes exhibit considerable antibacterial and antitumor activity [4–6]. As part of our continuous work, the title complex was synthesized and characterized by X-ray diffraction.

In the title crystal structure, the central copper ion is coordinated with two enolizated acylhydrazone ligands by NO bidentate donor sets and an additional coordination of ethanol (*cf*. the figure). The maximal two angles between the coordination atoms and Cu(II) ion are 174.36(10)° and 156.76(11)°, respectively. According to the Addison rule [5], the geometric index τ value is 0.293, indicating that the irregular coordination geometry of the copper ion should be described as a distorted square-pyramidal geometry. There also exist classical intermolecular N—H···O hydrogen bonds in the crystal.

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