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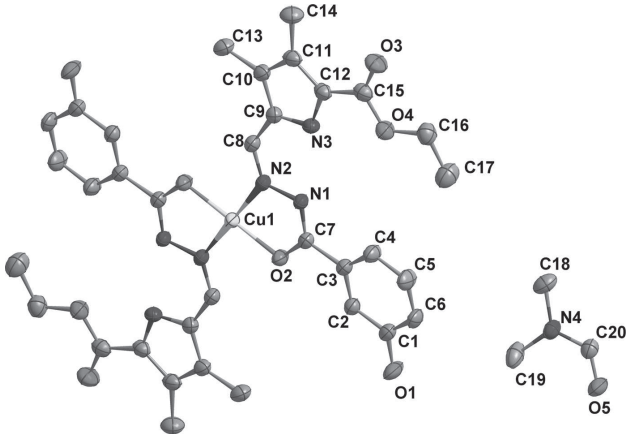
Crystal structure of bis-(*N'*-(5-ethoxycarbonyl-3,4-dimethyl-pyrrol-2-yl-methylidene)-3-hydroxybenzohydrazide-κ²*O*,*N*)copper(II) – dimethylformamide (1/2), $C_{40}H_{50}N_8O_{10}Cu$



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Abstract

$C_{40}H_{50}N_8O_{10}Cu$, triclinic, $P\bar{1}$ (no. 2), $a = 6.8237(9)$ Å, $b = 12.9229(17)$ Å, $c = 13.3275(18)$ Å, $\alpha = 104.949(2)°$, $\beta = 104.049(2)°$, $\gamma = 99.538(2)°$, $V = 1068.3(2)$ Å^3, $Z = 1$, $R_{gt}(F) = 0.0497$, $wR_{ref}(F^2) = 0.1343$, $T = 296(2)$ K.

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The crystal structure is shown in the figure. The asymmetric unit is labelled. 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 hydrazone ligand and the title complex were prepared according to the literature method [3].

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Table 1: Data collection and handling.

Crystal:	Brown block
Size:	0.23 × 0.20 × 0.19 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.58 mm^{-1}
Diffractometer, scan mode:	APEX Detector, φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5702, 3747, 0.021
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2778
$N(param)_{refined}$:	268
Programs:	SHELX [5], Bruker [6]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cu1	1.000000	0.500000	0.000000	0.0476(2)
O1	0.8905(5)	0.1187(2)	0.2604(2)	0.0776(8)
H1B	0.849800	0.084257	0.298261	0.116*
O2	0.9427(4)	0.40078(18)	0.07822(18)	0.0530(6)
O3	0.1448(4)	0.7726(2)	0.2918(2)	0.0793(8)
O4	0.2821(4)	0.6268(2)	0.2928(2)	0.0646(7)
O5	0.2396(4)	−0.0059(2)	0.6170(2)	0.0749(8)
N1	0.7235(4)	0.5099(2)	0.1258(2)	0.0473(7)
N2	0.8059(4)	0.5662(2)	0.0629(2)	0.0473(7)
N3	0.5015(4)	0.6659(2)	0.1603(2)	0.0487(7)
H3A	0.512050	0.607331	0.178387	0.058*
N4	0.2398(4)	0.1341(2)	0.5467(2)	0.0556(7)
C1	0.7859(6)	0.1982(3)	0.2540(3)	0.0552(9)
C2	0.8399(5)	0.2685(3)	0.1970(3)	0.0501(8)
H2A	0.944850	0.259578	0.164595	0.060*
C3	0.7400(5)	0.3517(3)	0.1877(3)	0.0488(8)
C4	0.5812(7)	0.3639(3)	0.2349(3)	0.0698(11)
H4A	0.512540	0.419565	0.229250	0.084*
C5	0.5268(8)	0.2932(3)	0.2898(4)	0.0842(14)
H5B	0.418140	0.300306	0.319809	0.101*
C6	0.6295(7)	0.2118(3)	0.3017(3)	0.0721(11)
H6A	0.593700	0.166229	0.341575	0.086*
C7	0.8059(5)	0.4250(3)	0.1266(2)	0.0450(7)
C8	0.7487(5)	0.6545(3)	0.0534(2)	0.0485(8)
H8A	0.808270	0.689189	0.011151	0.058*
C9	0.6072(5)	0.7064(3)	0.0987(3)	0.0491(8)
C10	0.5470(6)	0.8014(3)	0.0872(3)	0.0521(8)
C11	0.4008(6)	0.8178(3)	0.1430(3)	0.0547(9)
C12	0.3758(5)	0.7331(3)	0.1887(3)	0.0508(8)
C13	0.6296(6)	0.8747(3)	0.0283(3)	0.0669(10)
H13A	0.562447	0.934621	0.032360	0.100*

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
H13B	0.777124	0.903502	0.061368	0.100*
H13C	0.602206	0.832719	−0.046617	0.100*
C14	0.2889(7)	0.9089(3)	0.1510(4)	0.0770(12)
H14A	0.334958	0.956395	0.112160	0.116*
H14B	0.141621	0.877769	0.119799	0.116*
H14C	0.318591	0.950859	0.226060	0.116*
C15	0.2543(6)	0.7157(3)	0.2616(3)	0.0585(9)
C16	0.1776(6)	0.6043(4)	0.3703(3)	0.0716(11)
H16A	0.027731	0.585877	0.337727	0.086*
H16B	0.215738	0.668773	0.434099	0.086*
C17	0.2424(7)	0.5108(4)	0.4011(4)	0.0950(15)
H17A	0.175266	0.494100	0.452480	0.142*
H17B	0.203503	0.447365	0.337419	0.142*
H17C	0.390742	0.529922	0.433398	0.142*
C18	0.1934(7)	0.2401(3)	0.5521(4)	0.0776(12)
H18A	0.226998	0.263997	0.494456	0.116*
H18B	0.047935	0.233755	0.544329	0.116*
H18C	0.274473	0.293087	0.620992	0.116*
C19	0.3166(7)	0.0817(4)	0.4598(4)	0.0892(15)
H19A	0.331837	0.129083	0.415859	0.134*
H19B	0.449238	0.068471	0.490112	0.134*
H19C	0.219781	0.012839	0.415660	0.134*
C20	0.2073(5)	0.0837(3)	0.6171(3)	0.0583(9)
H20	0.155428	0.119374	0.671227	0.070*

Experimental details

The structure was solved by Direct Methods and refined with the SHELX crystallographic software package [5]. The hydrogen atoms were placed at calculated positions and refined as riding atoms with fixed isotropic displacement parameters.

Comment

Acylhydrazones are an important class of ligands in coordination chemistry and have been found extensive application in different fields [1]. Our previous work show that acylhydrazone ligands bearing pyrrole units and their complexes exhibit considerable antibacterial and antitumor activity [2–4]. As part of this study the title complex was synthesized and characterized by X-ray diffraction.

The asymmetric unit consists of one half of a copper(II) complex and one DMF molecule (*cf*. the figure). In the title crystal structure, the central copper ion is coordinated with two enolizated acylhydrazone ligands by NO bidentate donor sets, thus giving a distorted planar square coordination geometry. Two neighbouring DMF molecules link with the complex *via* pairs of O—H···O hydrogen bonds. N—H···O hydrogen bonds are also presented in the crystal. Cu–O and Cu–N bond lengths are all in the expected ranges [7].

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