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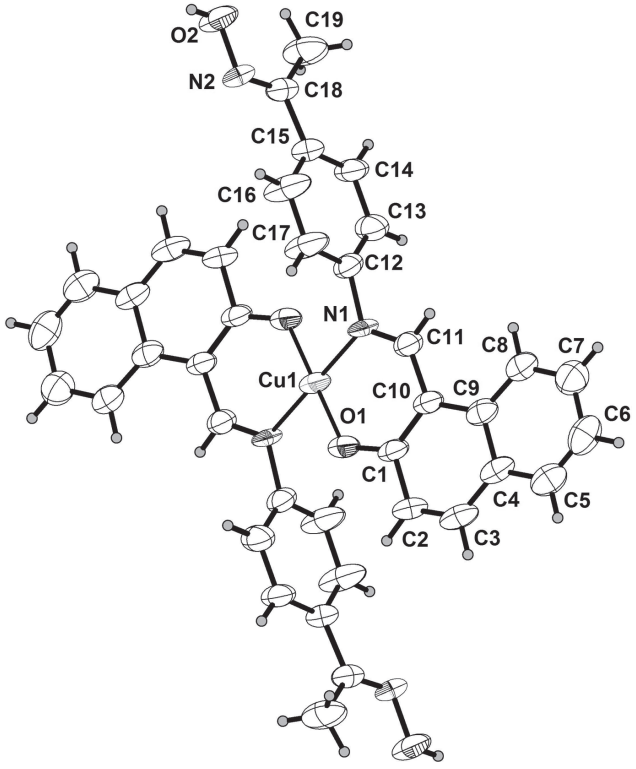
Synthesis and crystal structure of bis{1-(((4-(1-(hydroxyimino)ethyl)phenyl)imino)methyl)naphthalen-2-olato-κ^2*O*,*N*}copper(II), $C_{38}H_{30}CuN_4O_4$



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Abstract

$C_{38}H_{30}CuN_4O_4$, triclinic, $P\bar{1}$ (no. 2), $a = 8.3882(7)$ Å, $b = 8.5024(9)$ Å, $c = 11.3649(15)$ Å, $\alpha = 89.960(10)°$, $\beta = 69.689(10)°$, $\gamma = 80.269(8)°$, $Z = 1$, $V = 747.73(15)$ Å^3, $R_{gt}(F) = 0.0701$, $wR_{ref}(F^2) = 0.1308$, $T = 294.68(10)$ K.

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Table 1: Data collection and handling.

Crystal:	Plate, clear dark brown
Size:	0.33 × 0.06 × 0.05 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.78 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω-scans
$2\theta_{max}$, completeness:	52°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4922, 2937, 0.064
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1377
$N(param)_{refined}$:	216
Programs:	CrysAlis^{PRO} [1], SHELX [2]

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

Preparation of the ligand: 4-Amino acetophenone oxime was prepared by a similar method reported earlier [3–5]. 4-amino acetophenone oxime (148.2 mg, 1 mmol) in EtOH (5 mL) was added into 2-hydroxy-1-naphthaldehyde (175.2 mg, 1 mmol) in EtOH (5 mL). The reaction mixture was stirred under reflux at 328 K for 8 h. Then the mixture was filtered and washed, successively, after removing the solvent. The raw product was recrystallized with EtOH to get a yellow solid (yield 86.5%). Elemental analysis – Anal. calcd. for $C_{19}H_{16}N_2O_2$: C, 74.98%; H, 5.30%; N, 9.20%; Found: C, 75.02%; H, 5.28%; N, 9.16%.

Preparation of the copper(II) complex: A solution of copper(II) acetate monohydrate (2.1 mg, 0.01 mmol) in EtOH (2 mL) was added dropwise to a solution of 1-(4-(((2-hydroxynaphthalen-1-yl)methylene)amino)phenyl)ethan-1-one oxime (6.0 mg, 0.02 mmol) in EtOH (3 mL) at room temperature. The colour of the solution turned to brown immediately. After stirring for 1 h at room temperature, the mixture was filtered and the filtrate was allowed to stand at room temperature for two weeks. We obtained several clear dark brown block crystals. Elemental analysis – Anal. calcd. for $C_{38}H_{30}CuN_4O_4$: C, 68.10%; H, 4.51%; N, 8.36%; Found: C, 68.12%; H, 4.49%; N, 8.32%.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cu1	0.0000	0.5000	0.5000	0.0555(4)
O1	−0.1112(4)	0.4015(4)	0.6475(3)	0.0566(10)
O2	0.4070(5)	1.3655(4)	0.2875(4)	0.0761(13)
H2	0.3442	1.4536	0.3060	0.114*
N1	0.1372(5)	0.5887(5)	0.5868(4)	0.0483(12)
N2	0.3262(5)	1.2525(5)	0.3677(4)	0.0580(13)
C1	−0.0549(6)	0.3592(6)	0.7385(5)	0.0477(14)
C2	−0.1496(6)	0.2541(6)	0.8236(5)	0.0559(16)
H2A	−0.2402	0.2176	0.8093	0.067*
C3	−0.1066(7)	0.2086(7)	0.9242(5)	0.0629(17)
H3	−0.1721	0.1436	0.9795	0.076*
C4	0.0340(7)	0.2552(6)	0.9496(5)	0.0542(16)
C5	0.0784(8)	0.2034(7)	1.0528(6)	0.0670(18)
H5	0.0130	0.1382	1.1081	0.080*
C6	0.2135(9)	0.2460(8)	1.0734(6)	0.0731(19)
H6	0.2410	0.2094	1.1423	0.088*
C7	0.3136(8)	0.3444(7)	0.9928(6)	0.0721(18)
H7	0.4083	0.3718	1.0073	0.086*
C8	0.2719(7)	0.4001(6)	0.8932(6)	0.0586(16)
H8	0.3375	0.4679	0.8412	0.070*
C9	0.1304(7)	0.3574(6)	0.8659(5)	0.0500(14)
C10	0.0811(6)	0.4123(6)	0.7608(5)	0.0459(14)
C11	0.1602(6)	0.5334(6)	0.6875(5)	0.0493(15)
H11	0.2365	0.5775	0.7148	0.059*
C12	0.2192(7)	0.7244(6)	0.5376(5)	0.0494(14)
C13	0.3821(6)	0.7020(7)	0.4519(6)	0.0665(18)
H13	0.4483	0.5998	0.4331	0.080*
C14	0.4505(6)	0.8302(7)	0.3920(5)	0.0587(17)
H14	0.5623	0.8124	0.3333	0.070*
C15	0.3580(6)	0.9815(6)	0.4170(5)	0.0476(14)
C16	0.1977(7)	1.0046(7)	0.5116(5)	0.085(2)
H16	0.1358	1.1079	0.5365	0.102*
C17	0.1276(8)	0.8761(8)	0.5700(6)	0.084(2)
H17	0.0178	0.8935	0.6314	0.101*
C18	0.4272(7)	1.1166(7)	0.3418(5)	0.0513(15)
C19	0.5998(7)	1.0847(7)	0.2414(5)	0.088(2)
H19A	0.5998	1.0094	0.1785	0.132*
H19B	0.6852	1.0413	0.2769	0.132*
H19C	0.6264	1.1826	0.2039	0.132*

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Discussion

Transition metal complexes with oxime-type (-C = N—OR) ligands are an important research field at present [6–8]. And they have coordination abilities, which leads to potential application on photophysical properties materials [9, 10] and supramolecular architectures [11, 12] and so on.

We have designed and synthesized a new copper(II) complex. The complex is located at an inversion center. In the title complex, all bond lengths are in normal ranges. Cu1 atom is four-coordinated by two O atoms and two N atoms from two ligands. In the crystal structure, each molecule links two other adjacent ones into a 1D supramolecular structure through intermolecular O2–H2$\cdots$O1′ hydrogen bonds.

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