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Crystal structure of bis((1-(((4-(((benzyloxy)imino)methyl)phenyl)imino)methyl)naphthalen-2-yl)oxy- $\kappa^2 O, N$)copper(II), $C_{52}H_{42}CuN_4O_4$

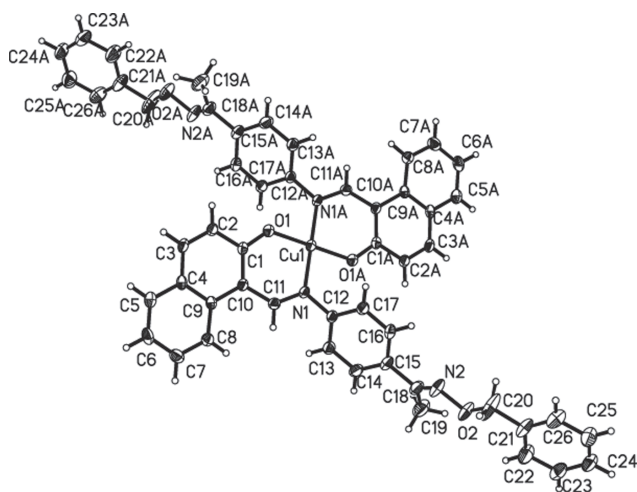


Table 1: Data collection and handling.

Crystal:	Block, clear light brown
Size:	0.24 × 0.15 × 0.14 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.56 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω -scans
$\theta_{\max}$, completeness:	26°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8438, 4218, 0.045
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2896
$N(\text{param})_{\text{refined}}$:	278
Programs:	CrysAlis ^{PRO} [1], SHELX [2]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cu1	0.5000	0.5000	0.5000	0.03460(18)
O1	0.46444(9)	0.7736(4)	0.47887(6)	0.0422(6)
O2	0.62223(13)	−0.0490(5)	0.74322(8)	0.0789(9)
N1	0.46770(11)	0.5559(4)	0.55213(7)	0.0341(6)
N2	0.60089(16)	0.0964(6)	0.71063(9)	0.0698(10)
C1	0.42302(13)	0.9011(6)	0.49173(9)	0.0336(7)
C2	0.40384(13)	1.0974(6)	0.46836(9)	0.0387(8)
H2	0.4204	1.1238	0.4447	0.046*
C3	0.36219(13)	1.2459(6)	0.47977(10)	0.0407(8)
H3	0.3522	1.3758	0.4644	0.049*
C4	0.33357(13)	1.2083(5)	0.51439(9)	0.0350(7)
C5	0.28910(14)	1.3604(6)	0.52522(10)	0.0459(9)
H5	0.2806	1.4933	0.5104	0.055*
C6	0.25800(15)	1.3174(7)	0.55703(11)	0.0520(10)
H6	0.2287	1.4201	0.5638	0.062*
C7	0.27061(15)	1.1176(7)	0.57931(10)	0.0511(9)
H7	0.2490	1.0861	0.6007	0.061*
C8	0.31459(14)	0.9681(6)	0.56979(9)	0.0410(8)
H8	0.3223	0.8360	0.5849	0.049*
C9	0.34831(12)	1.0098(5)	0.53772(9)	0.0330(7)
C10	0.39682(13)	0.8618(5)	0.52729(9)	0.0334(7)
C11	0.42365(13)	0.7000(6)	0.55586(9)	0.0363(7)
H11	0.4077	0.6970	0.5802	0.044*
C12	0.48849(13)	0.4214(6)	0.58685(9)	0.0353(7)
C13	0.44734(14)	0.2988(6)	0.60657(9)	0.0428(8)
H13	0.4053	0.3053	0.5976	0.051*
C14	0.46849(16)	0.1670(7)	0.63956(10)	0.0514(9)
H14	0.4404	0.0839	0.6523	0.062*
C15	0.53028(16)	0.1566(6)	0.65387(10)	0.0483(9)
C16	0.57119(15)	0.2822(6)	0.63410(10)	0.0495(9)
H16	0.6131	0.2785	0.6435	0.059*

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Abstract

$C_{52}H_{42}CuN_4O_4$, monoclinic, $I12/c1$ (no. 15), $a = 21.8573(13)$ Å, $b = 5.8605(4)$ Å, $c = 33.683(2)$ Å, $\beta = 97.428(5)^\circ$, $Z = 4$, $V = 4278.4(5)$ Å³, $R_{\text{gt}}(F) = 0.0554$, $wR_{\text{ref}}(F^2) = 0.1255$, $T = 293(2)$ K.

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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

Synthesis of the ligand HL: ({4-Amino}phenyl)ethanone *O*-benzyloxime was synthesized according to an analogous method reported previously in the literature [3].

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Table 2 (continued)

Atom	x	y	z	U _{iso} */U _{eq}
C17	0.55077(14)	0.4114(6)	0.60103(10)	0.0445(9)
H17	0.5789	0.4926	0.5881	0.053*
C18	0.55336(18)	0.0142(7)	0.68942(11)	0.0553(10)
C19	0.5222(2)	−0.2064(8)	0.69651(13)	0.0856(15)
H19A	0.5514	−0.3083	0.7110	0.128*
H19B	0.5064	−0.2738	0.6713	0.128*
H19C	0.4888	−0.1786	0.7118	0.128*
C20	0.6785(3)	0.0454(9)	0.76209(14)	0.116(2)
H20A	0.6704	0.1907	0.7742	0.139*
H20B	0.7062	0.0719	0.7423	0.139*
C21	0.7079(2)	−0.1151(8)	0.79360(12)	0.0717(13)
C22	0.6761(2)	−0.2899(9)	0.80915(13)	0.0837(15)
H22	0.6341	−0.3091	0.8008	0.100*
C23	0.7068(2)	−0.4371(9)	0.83726(13)	0.0842(15)
H23	0.6853	−0.5549	0.8477	0.101*
C24	0.7683(2)	−0.4106(10)	0.84966(13)	0.0838(15)
H24	0.7888	−0.5114	0.8681	0.101*
C25	0.7995(2)	−0.2355(10)	0.83485(14)	0.0861(15)
H25	0.8412	−0.2144	0.8438	0.103*
C26	0.7696(2)	−0.0887(9)	0.80656(13)	0.0817(14)
H26	0.7915	0.0286	0.7963	0.098*

To an ethanol solution (4 mL) of ({4-amino}phenyl) ethanone O-benzoyloxime (240.0 mg, 1.0 mmol) was added 2-hydroxy-1-naphthaldehyde (172.2 mg, 1.0 mmol). The mixture was stirred at 334 K for 18 h. After cooling to room temperature, the formed precipitate was filtered and washed successively with ethanol and ethanol/*n*-hexane (1:4), respectively. The product was dried in vacuo and purified by recrystallization from ethanol to yield 327.3 mg of solid. Yield, 79.4%. m.p. 400–402 K. Elemental analysis – Anal. calculated for C₂₅H₂₀N₂O₂ (%): C, 79.05; H, 5.30; N, 6.93. Found (%): C, 79.83; H, 5.01; N, 6.78.

Synthesis of the Cu(II) complex: A methanol solution (4 mL) of copper acetate monohydrate (1.3 mg, 0.007 mmol) was added dropwise to a dichloromethane solution (5 mL) of the aforementioned ligand (1.9 mg, 0.005 mmol) at room temperature. The solution turned to yellow immediately and the filtrate was allowed to stand at room temperature for about 1 week. Brown-red block crystals were obtained. Elemental analysis – Anal. calculated for C₅₂H₄₂CuN₄O₄ (%): C, 73.44; H, 4.98; N, 6.59. Found (%): C, 73.82; H, 4.66; N, 6.89.

Experimental details

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

Discussion

It is well known that some Schiff bases and their metal complexes are intensively investigated [4–9]. Especially the synthesis and crystal structure of oxime-type Schiff base

compounds and their derivatives [10–14]. These compounds are an important class of organic compounds because of their applications in many fields of organic, biological, analytical chemistry and material science [15–18]. Herein, a new copper(II) complex with Schiff base ligand (4-(((2-hydroxynaphthalen-1-yl)methylene)amino)benzaldehyde *O*-benzyl oxime) is reported.

The crystal structure is built up by a mononuclear Cu(II) complex, which constitutes the symmetric unit [Cu(L)₂]. As shown in the figure, the Cu(II) center is coordinated by a pair of N,O chelates from a pair of ligand units L[−]. The coordination geometry of the Cu(II) center can be described as a slightly distorted plane square involving N₂O₂ donor sets. In the crystal structure, the complex molecules are linked into an infinite 2D-layer supramolecular structure via C–H...π interactions.

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