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Synthesis and crystal structure of bis{((E)-((4-((E)-1-(benzyloxyimino)ethyl)phenyl)imino)methyl)-2-phenolato- $\kappa^2 N,O$ }copper(II), $C_{44}H_{38}CuN_4O_4$

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Abstract

$C_{44}H_{38}CuN_4O_4$, monoclinic, $P2_1/n$ (no. 14), $a = 8.0172(13)$ Å, $b = 10.9003(17)$ Å, $c = 42.154(8)$ Å, $\beta = 94.749(6)^\circ$, $Z = 4$, $V = 3671.2(11)$ Å³, $R_{gt}(F) = 0.0483$, $wR_{ref}(F^2) = 0.1072$, $T = 173(2)$ K.

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The molecular structure of the title complex is shown in the figure. Table 1 contains crystallographic data and Table 2

Table 1: Data collection and handling.

Crystal:	Dark brown block
Size:	0.19 × 0.18 × 0.15 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.65 mm ⁻¹
Diffractometer, scan mode:	Broker APEX-II, φ and ω -scans
θ_{max} , completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	26381, 6462, 0.063
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 4875
$N(param)_{refined}$:	480
Programs:	Broker programs [1], SHELX [2], OLEX2 [3]

contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

Synthesis of the ligand: All solvents and other reagents were of analytical grade. The synthesis of the ligand was prepared by a similar method reported earlier [4]. A solution containing 2-hydroxy-benzaldehyde (366.1 mg, 3 mmol) and 1-(4-aminophenyl)ethanone O-benzyl-oxime (720.9 mg, 3 mmol) in EtOH (10 mL) was heated at 328 K. 12 hours later, the mixture was filtered at room temperature. The product was

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

Atom	x	y	z	U_{iso}^*/U_{eq}
Cu1	0.50898(4)	0.65319(3)	0.25317(2)	0.02662(12)
N2	0.6454(3)	0.5881(2)	0.42746(6)	0.0386(7)
N1	0.3499(3)	0.5796(2)	0.28245(5)	0.0261(6)
N4	0.3664(3)	0.6789(2)	0.07976(6)	0.0366(7)
N3	0.6691(3)	0.7230(2)	0.22345(5)	0.0256(6)
O2	0.6973(3)	0.5620(2)	0.45956(5)	0.0494(6)
O1	0.3307(2)	0.68886(18)	0.22257(5)	0.0316(5)
O4	0.3087(3)	0.69629(19)	0.04760(5)	0.0434(6)
O3	0.6873(2)	0.61948(18)	0.28387(5)	0.0342(5)
C1	0.1994(3)	0.6192(2)	0.21637(7)	0.0260(7)
C2	0.1165(4)	0.6195(3)	0.18557(7)	0.0319(7)
H2	0.158656	0.669223	0.169551	0.038*
C3	−0.0248(4)	0.5492(3)	0.17810(8)	0.0367(8)
H3	−0.078316	0.551574	0.157142	0.044*
C4	−0.0895(4)	0.4749(3)	0.20097(8)	0.0387(8)
H4	−0.185738	0.425893	0.195678	0.046*
C5	−0.0127(4)	0.4734(3)	0.23111(7)	0.0320(7)
H5	−0.058560	0.424177	0.246823	0.038*
C6	0.1324(3)	0.5425(2)	0.23960(7)	0.0249(7)
C7	0.2055(4)	0.5357(2)	0.27154(7)	0.0273(7)
H7	0.142145	0.494866	0.286423	0.033*
C8	0.3999(4)	0.5586(3)	0.31541(7)	0.0274(7)
C9	0.3855(4)	0.4438(3)	0.32882(7)	0.0340(8)
H9	0.337616	0.378274	0.316319	0.041*
C10	0.4407(4)	0.4240(3)	0.36046(7)	0.0369(8)
H10	0.430392	0.344446	0.369309	0.044*
C11	0.5106(4)	0.5179(3)	0.37949(7)	0.0293(7)
C12	0.5200(4)	0.6342(3)	0.36556(7)	0.0324(7)
H12	0.563973	0.700775	0.378163	0.039*
C13	0.4675(4)	0.6542(3)	0.33428(7)	0.0309(7)
H13	0.477270	0.733694	0.325381	0.037*
C14	0.5743(4)	0.4959(3)	0.41312(7)	0.0346(8)
C15	0.5534(5)	0.3739(3)	0.42845(9)	0.0692(13)
H15A	0.593050	0.378736	0.451040	0.104*
H15B	0.618481	0.312092	0.417958	0.104*
H15C	0.434809	0.350863	0.426417	0.104*
C16	0.7784(4)	0.6708(3)	0.47320(8)	0.0523(10)
H16A	0.782586	0.665461	0.496695	0.063*
H16B	0.710758	0.743675	0.466474	0.063*
C17	0.9527(4)	0.6871(3)	0.46342(8)	0.0405(8)
C18	1.0906(5)	0.6438(3)	0.48225(9)	0.0522(10)
H18	1.075055	0.605866	0.502075	0.063*
C19	1.2492(5)	0.6552(4)	0.47253(11)	0.0623(11)
H19	1.342150	0.623907	0.485501	0.075*
C20	1.2741(5)	0.7110(4)	0.44446(11)	0.0608(11)
H20	1.384344	0.719883	0.438093	0.073*
C21	1.1401(5)	0.7546(4)	0.42527(10)	0.0618(11)
H21	1.157370	0.792725	0.405539	0.074*
C22	0.9797(5)	0.7426(3)	0.43489(9)	0.0514(10)
H22	0.887212	0.772910	0.421654	0.062*
C23	0.8215(4)	0.6886(2)	0.28942(7)	0.0268(7)
C24	0.9071(4)	0.6883(3)	0.31997(7)	0.0330(7)
H24	0.865977	0.639033	0.336198	0.040*
C25	1.0483(4)	0.7575(3)	0.32687(8)	0.0374(8)
H25	1.102722	0.755866	0.347764	0.045*

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C26	1.1129(4)	0.8299(3)	0.30365(8)	0.0368(8)
H26	1.210443	0.877946	0.308585	0.044*
C27	1.0341(4)	0.8310(3)	0.27366(7)	0.0312(7)
H27	1.079907	0.878676	0.257625	0.037*
C28	0.8864(3)	0.7633(2)	0.26588(7)	0.0250(7)
C29	0.8137(3)	0.7680(2)	0.23384(7)	0.0257(7)
H29	0.876769	0.807594	0.218695	0.031*
C30	0.6185(3)	0.7379(3)	0.19035(6)	0.0247(6)
C31	0.6290(4)	0.8495(3)	0.17507(7)	0.0324(7)
H31	0.676038	0.918401	0.186359	0.039*
C32	0.5713(4)	0.8611(3)	0.14335(7)	0.0338(7)
H32	0.579636	0.938298	0.133124	0.041*
C33	0.5013(3)	0.7625(3)	0.12614(7)	0.0272(7)
C34	0.4950(3)	0.6498(3)	0.14179(7)	0.0294(7)
H34	0.451411	0.580149	0.130373	0.035*
C35	0.5506(4)	0.6377(3)	0.17330(7)	0.0303(7)
H35	0.542794	0.560528	0.183555	0.036*
C36	0.4338(4)	0.7762(3)	0.09247(7)	0.0315(7)
C37	0.4447(5)	0.8957(3)	0.07543(8)	0.0595(11)
H37A	0.391446	0.887755	0.053743	0.089*
H37B	0.562447	0.918442	0.074543	0.089*
H37C	0.387178	0.959350	0.086810	0.089*
C38	0.2301(4)	0.5826(3)	0.03601(8)	0.0466(9)
H38A	0.301450	0.512526	0.043523	0.056*
H38B	0.222449	0.582442	0.012447	0.056*
C39	0.0585(4)	0.5656(3)	0.04698(8)	0.0418(9)
C44	0.0347(5)	0.5090(3)	0.07573(9)	0.0543(10)
H44	0.128705	0.479976	0.088793	0.065*
C43	−0.1249(6)	0.4943(4)	0.08562(10)	0.0646(11)
H43	−0.140125	0.454380	0.105205	0.077*
C42	−0.2614(5)	0.5379(4)	0.06688(12)	0.0694(13)
H42	−0.370692	0.528710	0.073715	0.083*
C41	−0.2402(5)	0.5945(4)	0.03848(12)	0.0662(13)
H41	−0.334355	0.624981	0.025717	0.079*
C40	−0.0803(5)	0.6071(3)	0.02839(9)	0.0510(10)
H40	−0.066225	0.644753	0.008460	0.061*

dried under vacuum and a yellow solid was obtained (yield 55.2%, m.p. 386–393 K). Elemental analysis: Anal. Calcd. for C₂₂H₂₀N₂O₂: C, 76.72%; H, 5.85%; N, 8.13%; Found: C, 76.32%; H, 5.97%; N, 8.01%. **Synthesis of the copper(II) complex:** A solution of copper(II) acetate monohydrate (2.0 mg, 10 mmol) in EtOH (2 mL) was added dropwise to the solution of the ligand (6.9 mg, 20 mmol) in CHCl₃ (4 mL) at room temperature. The color of the solution turned to brown, immediately. The mixture was filtered after being stirred for 2 h at room temperature and the filtrate was allowed to stand for 25 days. The solvent was partially evaporated and clear dark brown crystals were obtained. Anal. Calcd. for C₄₄H₃₈CuN₄O₄: C, 70.43%; H, 5.10%; N, 7.47%. Found: C, 70.34%; H, 5.26%; N, 7.22%.

Experimental details

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

Comment

Oximes are an important class of ligands due to their metal complexes, which have been widely studied and some studies on this subject are already available [4, 5]. Recently much attention is being focused on analytical reagents for the detection and determination of some metal ions [6], nonlinear optical materials field [7] and supramolecular architectures [8–12] etc.

The single crystal structure of the title complex was determined by X-ray crystallography. The bond lengths are in normal ranges. In the title complex, Cu1 is four-coordinated, with a N_2O_2 coordination sphere, and a planar arrangement. The Cu1–N1 bond length is 2.014(2) while the Cu1–N3 is 2.016(2). The Cu1–O1 bond length is 1.8849(18), while Cu1–O3 is 1.8836(19). The angles of N1–Cu1–O1 is 91.07(9) and the N3–Cu1–O1 is 89.00(9), while the N1–Cu1–O3 is 89.12(9) and the N3–Cu1–O3 is 90.83(9), respectively.

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