Jing Huang*, Jin-Xia Ma, Ping Wei and Ji-Xing Zhao*

Synthesis and crystal structure of bis{4-methyl-2-((E)-((4-((E)-1-(benzyloxyimino)ethyl)phenyl)imino)methyl)phenolato- $\kappa^3 N,O,O$ } copper(II), $C_{92}H_{84}Cu_2N_8O_8$

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Abstract

$C_{92}H_{84}Cu_2N_8O_8$, monoclinic, $P2_1/c$ (no. 14), $a = 18.315(2)$ Å, $b = 12.1533(12)$ Å, $c = 17.518(2)$ Å, $\beta = 105.289(4)^\circ$, $Z = 2$, $V = 3761.3(7)$ Å³, $R_{gt}(F) = 0.0658$, $wR_{ref}(F^2) = 0.1809$, $T = 173(2)$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

Synthesis of the ligand: Synthesis of the ligand was prepared by a similar method reported earlier [4, 5]. 2-Hydroxy-5-methyl-benzaldehyde (408.2 mg, 3 mmol) in EtOH (5 mL) was added slowly to a solution of 1-(4-aminophenyl)ethanone O-

Table 1: Data collection and handling.

Crystal:	Dark brown block
Size:	$0.19 \times 0.17 \times 0.14$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.63 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	25° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	18776, 6610, 0.074
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 5104
$N(\text{param})_{\text{refined}}$:	500
Programs:	Bruker programs [1], SHELX [2], OLEX2 [3]

benzyl-oxime (720.9 mg, 3 mmol) in EtOH (5 mL) kept refluxing for 12 h at 328 K. The mixture was filtered at room temperature. The product was dried under vacuum and a red solid was obtained (yield 45.2%, M. pt. 372–381 K).

Synthesis of the copper(II) complex: The copper(II) acetate monohydrate (2.0 mg, 10 mmol) in EtOH (2 mL) was added slowly to a solution of ligand (7.2 mg, 20 mmol) in $CHCl_3$ (4 mL) at room temperature. The mixture was filtered after being stirred for 2 h at room temperature and stand for 19 days at room temperature. Several clear dark brown crystals were obtained. Anal. Calcd. for $C_{92}H_{84}Cu_2N_8O_8$: C, 70.98%; H, 5.44%; N, 7.20%. Found: C, 71.31%; H, 5.56%; N, 7.06%.

Experimental details

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

Comment

Oxime-type compounds are prepared by condensation reaction of aldehydes or ketones with hydroxylamine [6]. The oxime ligands form stable complexes with transition metals through the nitrogen and oxygen atoms [7–10]. There is an interest on such complexes, for their applications [11]. For example: they have been used to identify metal ions [12, 13], have been used in the field of fluorescence [14, 15] and supramolecular architectures [16–18] etc.

The crystal structure of the title compound is only built up by the $C_{92}H_{84}Cu_2N_8O_8$ complexes. In the title complex, Cu1 atom is five-coordinate, with a N_2O_3 coordination

***Corresponding authors:** Jing Huang, School of Chemical Engineering, Northwest Minzu University, Lanzhou 730030, People's Republic of China; and Key Laboratory for Utility of Environment-Friendly Composite Materials and Biomass in Universities of Gansu Province, Lanzhou 730124, People's Republic of China, e-mail: shiyihuangjing@126.com; and Ji-Xing Zhao, Analysis and Testing Center, Shihezi University, Shihezi 832003, People's Republic of China, e-mail: zhaojixing_2016@126.com

Jin-Xia Ma and Ping Wei: School of Chemical and Biological Engineering, Lanzhou Jiaotong University, Lanzhou 730070, P.R. China

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
Cu1	0.51582(2)	0.40934(4)	0.43624(3)	0.01703(18)
N1	0.88211(18)	0.7659(3)	0.5708(2)	0.0296(9)
N2	0.57912(16)	0.5172(2)	0.39065(18)	0.0168(7)
N3	0.46621(16)	0.2792(2)	0.47603(18)	0.0165(7)
N4	0.19777(17)	−0.0318(3)	0.2894(2)	0.0254(8)
O1	0.95701(15)	0.7992(2)	0.60645(19)	0.0327(8)
O2	0.46796(15)	0.3605(2)	0.33266(15)	0.0241(6)
O3	0.13191(16)	−0.0770(2)	0.23718(18)	0.0329(8)
O4	0.56920(14)	0.4479(2)	0.54208(15)	0.0189(6)
C1	1.0753(3)	0.9722(4)	0.5974(3)	0.0424(13)
H1	1.053258	0.953490	0.543627	0.051*
C2	1.1478(3)	1.0156(4)	0.6189(3)	0.0444(13)
H2	1.174904	1.027722	0.580315	0.053*
C3	1.1798(2)	1.0410(4)	0.6973(3)	0.0346(11)
H3	1.229441	1.070906	0.712939	0.042*
C4	1.1404(3)	1.0232(4)	0.7528(3)	0.0406(12)
H4	1.162937	1.040481	0.806796	0.049*
C5	1.0677(3)	0.9800(4)	0.7301(3)	0.0356(11)
H5	1.040744	0.967281	0.768746	0.043*
C6	1.0344(2)	0.9554(4)	0.6517(3)	0.0306(10)
C7	0.9540(2)	0.9132(4)	0.6265(3)	0.0406(13)
H7A	0.929414	0.921790	0.670122	0.049*
H7B	0.924444	0.955164	0.580144	0.049*
C8	0.8773(2)	0.6630(3)	0.5528(2)	0.0253(9)
C9	0.8003(2)	0.6242(3)	0.5119(2)	0.0223(9)
C10	0.7775(2)	0.5161(3)	0.5162(2)	0.0247(9)
H10	0.812583	0.464358	0.545900	0.030*
C11	0.7050(2)	0.4814(3)	0.4785(2)	0.0211(9)
H11	0.690959	0.406882	0.483097	0.025*
C12	0.6524(2)	0.5553(3)	0.4338(2)	0.0197(8)
C13	0.6746(2)	0.6650(3)	0.4305(2)	0.0242(9)
H13	0.639265	0.716967	0.401473	0.029*
C14	0.7463(2)	0.6987(3)	0.4682(2)	0.0246(9)
H14	0.759882	0.773654	0.464800	0.030*
C15	0.5533(2)	0.5570(3)	0.3199(2)	0.0191(8)
H15	0.581635	0.614816	0.305096	0.023*
C16	0.4861(2)	0.5224(3)	0.2618(2)	0.0193(8)
C17	0.4620(2)	0.5853(3)	0.1913(2)	0.0190(8)
H17	0.488188	0.651303	0.186247	0.023*
C18	0.4016(2)	0.5531(3)	0.1299(2)	0.0222(9)
C19	0.3664(2)	0.4544(3)	0.1386(2)	0.0247(9)
H19	0.325311	0.430112	0.096607	0.030*
C20	0.3884(2)	0.3902(3)	0.2056(2)	0.0220(9)
H20	0.362786	0.323033	0.208397	0.026*
C21	0.4487(2)	0.4230(3)	0.2702(2)	0.0172(8)
C22	0.3744(2)	0.6228(4)	0.0566(2)	0.0318(10)
H22A	0.388337	0.587543	0.012181	0.048*
H22B	0.397897	0.695737	0.065987	0.048*
H22C	0.319219	0.630478	0.044111	0.048*
C23	0.0413(2)	−0.2238(4)	0.3422(3)	0.0386(12)
H23	−0.000210	−0.218189	0.296503	0.046*
C24	0.0275(3)	−0.2424(4)	0.4156(3)	0.0488(14)
H24	−0.022958	−0.249865	0.419633	0.059*
C25	0.0866(3)	−0.2498(4)	0.4817(3)	0.0508(14)
H25	0.077461	−0.262744	0.531860	0.061*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C26	0.1599(3)	−0.2385(4)	0.4752(3)	0.0480(14)
H26	0.200976	−0.242869	0.521442	0.058*
C27	0.1743(3)	−0.2211(4)	0.4029(3)	0.0387(12)
H27	0.225035	−0.214424	0.399530	0.046*
C28	0.1145(2)	−0.2132(3)	0.3345(3)	0.0311(10)
C29	0.1296(2)	−0.1923(3)	0.2557(3)	0.0336(11)
H29A	0.178649	−0.226298	0.255330	0.040*
H29B	0.089685	−0.228394	0.214014	0.040*
C30	0.2069(2)	0.0685(3)	0.2742(2)	0.0246(9)
C31	0.1551(3)	0.1333(4)	0.2082(3)	0.0471(14)
H31A	0.117893	0.083590	0.174855	0.071*
H31B	0.185076	0.168337	0.176109	0.071*
H31C	0.128847	0.189884	0.230653	0.071*
C32	0.2751(2)	0.1232(3)	0.3262(2)	0.0202(9)
C33	0.2770(2)	0.2356(3)	0.3393(2)	0.0250(9)
H33	0.234183	0.278830	0.314125	0.030*
C34	0.3394(2)	0.2866(3)	0.3879(2)	0.0233(9)
H34	0.339408	0.363953	0.395534	0.028*
C35	0.4022(2)	0.2244(3)	0.4257(2)	0.0187(8)
C36	0.4012(2)	0.1110(3)	0.4136(2)	0.0221(9)
H36	0.444121	0.067941	0.438722	0.027*
C37	0.3379(2)	0.0608(3)	0.3648(2)	0.0245(9)
H37	0.337327	−0.016756	0.357686	0.029*
C38	0.4947(2)	0.2345(3)	0.5451(2)	0.0179(8)
H38	0.470539	0.170227	0.557325	0.021*
C39	0.5595(2)	0.2737(3)	0.6045(2)	0.0168(8)
C40	0.5866(2)	0.2063(3)	0.6717(2)	0.0205(8)
H40	0.560890	0.139408	0.675453	0.025*
C41	0.6488(2)	0.2341(3)	0.7321(2)	0.0216(9)
C42	0.6841(2)	0.3339(3)	0.7254(2)	0.0213(9)
H42	0.727249	0.355051	0.766175	0.026*
C43	0.6586(2)	0.4029(3)	0.6615(2)	0.0194(8)
H43	0.684623	0.469844	0.658990	0.023*
C44	0.5944(2)	0.3758(3)	0.5998(2)	0.0168(8)
C45	0.6764(2)	0.1618(4)	0.8038(2)	0.0287(10)
H45A	0.643612	0.171634	0.839410	0.043*
H45B	0.728397	0.182176	0.831463	0.043*
H45C	0.675010	0.084692	0.787200	0.043*
C46	0.9433(2)	0.5858(4)	0.5686(3)	0.0385(12)
H46A	0.969243	0.592987	0.526571	0.058*
H46B	0.925367	0.510074	0.569962	0.058*
H46C	0.978521	0.603957	0.619682	0.058*

environment. Bond lengths and angles are all in the expected ranges.

Each, almost planar complex and a inversion symmetry relate one are stacked pairwise (*cf.* the figure) in the title crystal structure.

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