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Crystal structure of bis{ μ_2 -bis{((((1-methoxyimino)ethyl)phenyl)imino)methyl)-2-phenolato- $\kappa^3 N, O:O$ }copper(II)}, $C_{68}H_{68}Cu_2N_8O_8$

<https://doi.org/10.1515/ncrs-2019-0062>

Received January 20, 2019; accepted March 29, 2019; available online May 8, 2019

Abstract

$C_{68}H_{68}Cu_2N_8O_8$, monoclinic, $P2_1/c$, $a = 16.241(3)$ Å, $b = 12.1604(19)$ Å, $c = 15.531(3)$ Å, $\beta = 96.364(3)^\circ$, $Z = 2$, $V = 3048.3(9)$ Å³, $R_{gt}(F) = 0.0497$, $wR_{ref}(F^2) = 0.1263$, $T = 296(2)$ K.

CCDC no.: 1906567

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: All solvents and other reagents were of analytical grade and the ligand was prepared by modification of the reported method [4–6]. To an ethanolic solution (8 mL) of 1-(4-aminophenyl)ethan-1-one O-ethyl oxime

Table 1: Data collection and handling.

Crystal:	Dark brown block
Size:	$0.27 \times 0.26 \times 0.22$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.76 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	27.6° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	20682, 7046, 0.061
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4271
$N(\text{param})_{\text{refined}}$:	392
Programs:	Bruker programs [1], SHELX [2], OLEX2 [3]

(328.0 mg, 2 mmol) was added an ethanolic solution (5 mL) of salicylaldehyde (244.1 mg, 2 mmol). After stirring and refluxing for 9 hours at 328 K, the mixture was filtered at room temperature. The product was dried under vacuum and a yellow solid was obtained (yield 67.3%, m.p. 389–397 K). Elemental analysis: Anal. Calcd. For $C_{17}H_{18}N_2O_2$: C, 72.32%; H, 6.43%; N, 9.92%; Found: C, 72.61%; H, 6.71%; N, 9.71%.

Synthesis of the copper(II) complex: To an ethanolic solution (2 mL) of copper(II) acetate monohydrate (2.0 mg, 10 mmol) was added an chloroform solution (4 mL) of ligand (5.7 mg, 20 mmol) at room temperature. The mixture was filtered after being stirred for 2 h. Then the filtrate was allowed to stand for 12 days. Several clear dark brown crystals were obtained. Anal. Calcd. for $C_{68}H_{68}Cu_2N_8O_8$: C, 65.21%; H, 5.47%; N, 8.95%. Found: C, 65.45%; H, 5.76%; N, 8.72%.

Experimental details

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

Comment

Schiff bases have been reported as an important class of ligands [7]. Their metal complexes have been widely studied [8–11]. Our research group has focus on the study of Schiff base and their complexes. Recently much attention is being focused on analytical reagents for the detection and determination of some metal ions [12, 13], specific properties [14, 15] and supramolecular architectures [16–18] etc.

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
Cu1	0.50267(2)	0.58933(3)	0.58334(3)	0.03836(13)
N1	0.44797(13)	0.71956(17)	0.51555(16)	0.0384(6)
N2	0.07705(19)	0.8166(3)	0.5883(3)	0.0961(13)
N3	0.57240(13)	0.48781(18)	0.66722(17)	0.0383(6)
N4	0.95590(16)	0.4472(3)	0.6188(2)	0.0697(9)
O1	0.57367(11)	0.56515(14)	0.49471(14)	0.0409(5)
O2	0.00108(16)	0.8596(3)	0.6081(3)	0.1143(13)
O3	0.43190(12)	0.61882(16)	0.66898(15)	0.0491(6)
O4	1.03612(15)	0.4091(2)	0.6061(2)	0.0849(9)
C1	0.60360(17)	0.6427(2)	0.4473(2)	0.0374(7)
C2	0.67952(18)	0.6268(2)	0.4140(2)	0.0491(8)
H2	0.709734	0.563249	0.428737	0.059*
C3	0.71048(19)	0.7021(3)	0.3604(2)	0.0563(9)
H3	0.760915	0.688749	0.339254	0.068*
C4	0.6676(2)	0.7979(3)	0.3373(2)	0.0595(10)
H4	0.688371	0.848643	0.300479	0.071*
C5	0.5943(2)	0.8163(2)	0.3698(2)	0.0525(9)
H5	0.565250	0.880479	0.354214	0.063*
C6	0.56080(17)	0.7424(2)	0.4258(2)	0.0390(7)
C7	0.48410(17)	0.7705(2)	0.4570(2)	0.0425(7)
H7	0.457126	0.832379	0.432589	0.051*
C8	0.37071(17)	0.7648(2)	0.5364(2)	0.0417(7)
C9	0.36063(18)	0.8760(2)	0.5518(2)	0.0477(8)
H9	0.404643	0.924257	0.548835	0.057*
C10	0.28483(19)	0.9151(2)	0.5715(2)	0.0540(9)
H10	0.279098	0.989699	0.582439	0.065*
C11	0.21759(19)	0.8467(3)	0.5755(2)	0.0525(9)
C12	0.22922(19)	0.7352(3)	0.5580(2)	0.0586(10)
H12	0.184701	0.687181	0.558600	0.070*
C13	0.30369(18)	0.6947(2)	0.5401(2)	0.0516(9)
H13	0.309651	0.619833	0.530264	0.062*
C14	0.1354(2)	0.8868(3)	0.5964(3)	0.0658(11)
C15	0.1242(3)	1.0016(3)	0.6272(3)	0.0998(16)
H15A	0.126060	1.051828	0.579741	0.150*
H15B	0.167777	1.019065	0.672048	0.150*
H15C	0.071639	1.007782	0.649596	0.150*
C16	−0.0622(3)	0.7776(4)	0.5916(5)	0.167(3)
H16A	−0.062811	0.750697	0.532728	0.201*
H16B	−0.050368	0.716126	0.630769	0.201*
C17	−0.1400(3)	0.8224(5)	0.6032(5)	0.182(4)
H17A	−0.144886	0.829370	0.664008	0.273*
H17B	−0.182932	0.774754	0.577155	0.273*
H17C	−0.145467	0.893514	0.576384	0.273*
C18	0.33830(19)	0.5704(3)	0.7675(2)	0.0497(8)
H18	0.304439	0.629353	0.748822	0.060*
C19	0.3157(2)	0.5038(3)	0.8322(2)	0.0578(9)
H19	0.267507	0.519038	0.857078	0.069*
C20	0.3638(2)	0.4137(3)	0.8611(3)	0.0632(10)
H20	0.347821	0.368299	0.904491	0.076*
C21	0.4353(2)	0.3932(3)	0.8245(2)	0.0541(9)
H21	0.467694	0.333079	0.843671	0.065*
C22	0.46104(17)	0.4604(2)	0.7589(2)	0.0410(7)
C23	0.41170(17)	0.5518(2)	0.7287(2)	0.0404(7)
C24	0.53952(17)	0.4397(2)	0.7298(2)	0.0424(8)
H24	0.571236	0.384940	0.759136	0.051*

Table 2 (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C25	0.65633(17)	0.4564(2)	0.6575(2)	0.0423(7)
C26	0.68336(18)	0.3500(2)	0.6717(2)	0.0497(8)
H26	0.646361	0.296424	0.686094	0.060*
C27	0.76483(18)	0.3216(3)	0.6648(2)	0.0565(9)
H27	0.781884	0.249368	0.675173	0.068*
C28	0.82126(18)	0.3985(3)	0.6427(2)	0.0513(8)
C29	0.79328(18)	0.5062(3)	0.6282(2)	0.0546(9)
H29	0.830210	0.559697	0.613516	0.066*
C30	0.71214(18)	0.5352(2)	0.6351(2)	0.0486(8)
H30	0.694841	0.607414	0.624875	0.058*
C31	0.9087(2)	0.3672(3)	0.6327(3)	0.0608(10)
C32	0.9354(2)	0.2501(3)	0.6379(4)	0.1046(18)
H32A	0.994038	0.245849	0.634987	0.157*
H32B	0.906886	0.209848	0.590383	0.157*
H32C	0.922447	0.218946	0.691569	0.157*
C33	1.0885(2)	0.5029(4)	0.6069(3)	0.0847(14)
H33A	1.060748	0.560113	0.571086	0.102*
H33B	1.138918	0.483465	0.582446	0.102*
C34	1.1097(3)	0.5453(5)	0.6953(4)	0.127(2)
H34A	1.059877	0.564256	0.719844	0.191*
H34B	1.143955	0.609420	0.693492	0.191*
H34C	1.139184	0.489854	0.730353	0.191*

The single crystal structure of the title complex was determined by X-ray crystallography. This crystal structure of the title complex is built up by the C₆₈H₆₈Cu₂N₈O₈ complex (*cf.* the figure). In the title complex, Cu1 atom is five-coordinate, with a N₂O₃ coordination environment with three O atoms (O1, O3 and O1ⁱ) and two N atoms (N1 and N3). The Cu1—O1 bond length is 1.914(2) Å and the Cu1—O3 is 1.886(2) Å and Cu1—O1ⁱ is 2.490 Å, respectively. The Cu1—N1 bond length is 2.049(2) Å and the Cu1—N3 is 2.044(2) Å, respectively.

Geometric parameters are all in the expected ranges for such a complex.

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