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Crystal structure of ((5,5'-dimethoxy-2,2'-(1,2-phenylenebis(nitrilomethyldiyn))diphenolato- $\kappa^4 O,N,O',N'$)copper(II), $C_{22}H_{18}N_2CuO_4$

Table 1: Data collection and handling.

Crystal:	Blue block
Size:	0.12 × 0.10 × 0.08 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.24 mm ⁻¹
Diffractometer, scan mode:	APEX2, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	24774, 3205, 0.029
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2888
$N(\text{param})_{\text{refined}}$:	264
Programs:	Bruker [1], SHELX [2, 3]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cu1	0.55216(2)	0.70941(5)	0.66651(2)	0.03281(10)
N1	0.59322(10)	0.9898(3)	0.71656(7)	0.0324(4)
N2	0.46405(10)	0.6937(3)	0.72637(7)	0.0321(4)
O1	0.88902(10)	1.0285(3)	0.51769(7)	0.0503(4)
O2	0.64259(10)	0.7268(3)	0.61170(7)	0.0478(4)
O3	0.51050(9)	0.4293(3)	0.62042(6)	0.0395(3)
O4	0.32969(11)	-0.2400(3)	0.55813(8)	0.0490(4)
C1	0.88868(18)	0.8170(5)	0.47759(12)	0.0603(7)
H1A	0.8878	0.6655	0.5016	0.090*
H1B	0.9387	0.8196	0.4537	0.090*
H1C	0.8393	0.8231	0.4503	0.090*
C2	0.76391(13)	0.8677(4)	0.56504(10)	0.0393(5)
H2	0.7596	0.7358	0.5369	0.047*
C3	0.82867(13)	1.0382(4)	0.56081(9)	0.0382(5)
C4	0.83739(15)	1.2385(4)	0.60250(11)	0.0448(5)
H4	0.8811	1.3542	0.5991	0.054*
C5	0.78127(15)	1.2609(4)	0.64774(11)	0.0436(5)
H5	0.7873	1.3939	0.6754	0.052*
C6	0.71327(12)	1.0892(4)	0.65473(9)	0.0347(4)
C7	0.70367(12)	0.8893(4)	0.61146(9)	0.0353(4)
C8	0.65797(13)	1.1290(4)	0.70358(9)	0.0364(4)
H8	0.6688	1.2676	0.7288	0.044*
C9	0.54211(12)	1.0421(4)	0.76736(9)	0.0334(4)
C10	0.55541(14)	1.2327(4)	0.81004(10)	0.0398(5)
H10	0.6019	1.3386	0.8070	0.048*
C11	0.49993(15)	1.2664(4)	0.85712(11)	0.0450(5)
H11	0.5091	1.3956	0.8855	0.054*
C12	0.43101(15)	1.1096(5)	0.86228(10)	0.0472(5)

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Abstract

$C_{22}H_{18}N_2CuO_4$, monoclinic, $P2_1/n$ (no. 14), $a = 15.7861(11)$ Å, $b = 5.3211(4)$ Å, $c = 21.6563(16)$ Å, $\beta = 92.129(4)^\circ$, $V = 1817.9(2)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0254$, $wR_{\text{ref}}(F^2) = 0.0765$, $T = 298$ 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.

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H12	0.3935	1.1343	0.8939	0.057*
C13	0.41753(14)	0.9165(4)	0.82080(10)	0.0429(5)
H13	0.3715	0.8097	0.8250	0.051*
C14	0.47201(12)	0.8793(4)	0.77270(9)	0.0338(4)
C15	0.40166(12)	0.5323(4)	0.72438(9)	0.0349(4)
H15	0.3624	0.5443	0.7552	0.042*
C16	0.38841(12)	0.3424(4)	0.68003(9)	0.0333(4)
C17	0.44322(12)	0.2969(4)	0.63026(9)	0.0337(4)
C18	0.42381(13)	0.0969(4)	0.58957(9)	0.0380(5)
H18	0.4600	0.0630	0.5577	0.046*
C19	0.35265(13)	−0.0498(4)	0.59576(9)	0.0384(5)
C20	0.29804(13)	−0.0041(5)	0.64504(10)	0.0435(5)
H20	0.2501	−0.1027	0.6496	0.052*
C21	0.31625(13)	0.1839(4)	0.68523(10)	0.0404(5)
H21	0.2803	0.2111	0.7176	0.048*
C24	0.38329(17)	−0.2969(5)	0.50765(11)	0.0518(6)
H24A	0.3870	−0.1525	0.4813	0.078*
H24B	0.3598	−0.4354	0.4844	0.078*
H24C	0.4389	−0.3406	0.5236	0.078*

Source of material

The educt, 6,6'-((1,2-phenylenebis(azanylylidene))bis(methanylylidene))bis(3-methoxyphenol) ligand was purchased from Acros Ltd. and used without further purification, the other reagents were commercially available and used as purchased [1–3]. A mixture of CuCl₂ (53.78 mg, 0.4 mmol), Schiff base ligand (49.64 mg, 0.4 mmol) and distilled water (10 mL) was sealed in a 23 mL Teflon-lined steel vessel (solution pH 2.5) and heated at 160 °C for 72 h, and then cooled to room temperature at a rate of 0.05 °C min^{−1}. The resulting light turquoise block crystals of the compound were obtained and washed with distilled water. The yield is ca. 32%. IR(KBr): 3256(s), 1597(s), 1518(s), 1497(s), 1448(s), 1356(s), 1245(s), 1174(s), 1113(s), 1073(s), 1005(s), 924(s), 894(s), 835(m), 773(m), 716(m), 619(m), 493(m) cm^{−1}.

Experimental details

All H atoms were placed geometrically and treated as riding on their parent atoms, with C–H 0.96, with $U_{iso}(H) = 1.5U_{eq}(C)$.

Comment

There has been considerable interest in Schiff base ligands and their metal complexes due to their variety of applications in chemistry, biology, physics and in catalysis [4–6].

The compound crystallizes in the monoclinic space group $P2_1/n$ with four formula units in the unit cell. As shown in the figure, one formula unit consists of one Schiff base ligand and one Cu atom. The Cu1 ion is in a slightly distorted square coordination environment, with two nitrogen atoms and two oxygen atoms from the Schiff base title ligand. The bond length of Cu1–N1 and Cu1–N2 is 1.939 Å and 1.936 Å, while the bond length of Cu1–O2 and Cu1–O3 is 1.893 Å and 1.899 Å. The geometric parameters are all in the expected ranges for such complexes [7–9].

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