Crystal structure of bis[*N*-cyclohexyl-4-nitrosalicylaldiminato]copper(II), $Cu(C_{13}H_{15}N_2O_3)_2$

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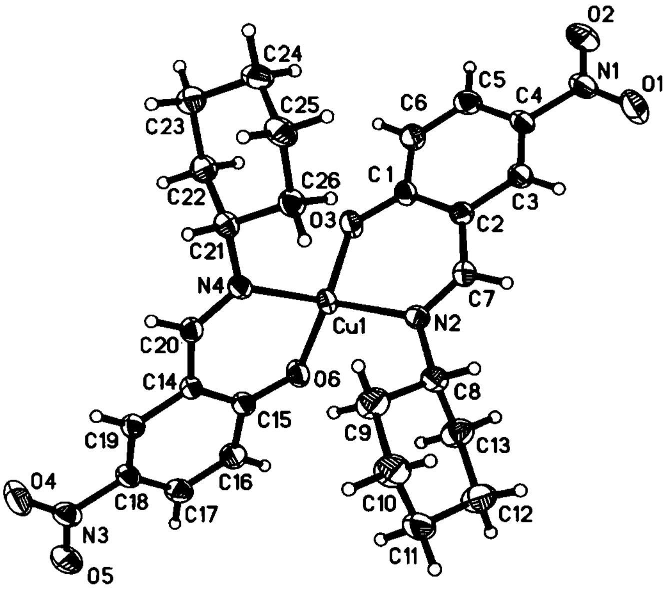

Abstract

$C_{26}H_{30}CuN_4O_6$, triclinic, $P\bar{1}$ (no. 2), $a = 10.765(2)$ Å, $b = 11.113(2)$ Å, $c = 12.441(2)$ Å, $\alpha = 113.746(2)°$, $\beta = 104.388(2)°$, $\gamma = 94.983(2)°$, $V = 1290.0$ Å^3, $Z = 2$, $R_{gt}(F) = 0.066$, $wR_{ref}(F^2) = 0.123$, $T = 298$ K.

Source of material

Reagents and solvents used were of commercially available quality. 5-Nitro-2-hydroxybenzaldehyde (0.2 mmol, 33.5 mg), cyclohexylamine (0.2 mmol, 19.7 mg) and $Cu(CH_3COO)_2 \cdot H_2O$ (0.1 mmol, 19.9 mg) were dissolved in EtOH (30 ml). The mixture was stirred at reflux for about 30 min and cooled to room temperature. After keeping the filtrate in dark for 12 days, blue block crystals were generated.

Discussion

Copper-containing metalloproteins are widely found in nature. Among them are two classes of compounds responsible for rapid intra- and intermolecular electron transfer in biological systems: those containing mononuclear blue copper sites [1-4] and those containing dinuclear CuA centers [5-7]. The mononuclear copper center is found in plastocyanin, azurin, stellacyanin, and the multicopper oxidases. The CuA center found in nitrous oxide reductase and cytochrome *c* oxidase is a delocalized mixed-valence dicopper center [8,9].

The title compound is a mononuclear copper(II) complex, which is structurally similar to the complexes reported earlier [10,11]. The Cu1 atom is four-coordinated by two N and two O atoms from two Schiff bases in a tetrahedral environment, which is different from the square planar arrangement in the brown form of the *N*-cyclohexylsalicylaldiminato copper(II) complex reported a few years ago [12]. All the bond lengths subtended at the Cu1 atom are comparable to the corresponding values observed in other Schiff base copper(II) complexes [12-14].

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Table 1. Data collection and handling.

Crystal:	blue block, size 0.07 × 0.10 × 0.18 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	8.95 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	54°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10917, 5519
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3974
$N(param)_{refined}$:	334
Program:	SHELXTL [15]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(3)	2*i*	0.5393	0.8798	0.3701	0.052
H(5)	2*i*	0.5114	0.5109	0.1107	0.061
H(6)	2*i*	0.7038	0.4970	0.2278	0.061
H(7)	2*i*	0.7115	0.9872	0.5484	0.052
H(8)	2*i*	0.8285	1.1071	0.7323	0.052
H(9A)	2*i*	1.0463	1.0184	0.8317	0.079
H(9B)	2*i*	0.9086	0.9977	0.8508	0.079
H(10A)	2*i*	0.9326	1.2282	0.9627	0.086
H(10B)	2*i*	1.0556	1.1849	1.0234	0.086
H(11A)	2*i*	1.1813	1.2659	0.9323	0.081
H(11B)	2*i*	1.1164	1.3812	1.0034	0.081
H(12A)	2*i*	1.0975	1.3630	0.8038	0.082
H(12B)	2*i*	0.9589	1.3416	0.8209	0.082
H(13A)	2*i*	0.9532	1.1728	0.6294	0.075
H(13B)	2*i*	1.0748	1.1299	0.6933	0.075
H(16)	2*i*	1.3843	0.9465	0.6683	0.069
H(17)	2*i*	1.5646	0.9437	0.8087	0.067
H(19)	2*i*	1.3373	0.7439	0.9189	0.051
H(20)	2*i*	1.1148	0.6705	0.8269	0.048
H(21)	2*i*	0.9105	0.6076	0.7876	0.049
H(22A)	2*i*	0.9363	0.4344	0.6180	0.058
H(22B)	2*i*	0.8400	0.4752	0.5273	0.058
H(23A)	2*i*	0.7648	0.3703	0.6806	0.067
H(23B)	2*i*	0.7257	0.2928	0.5359	0.067
H(24A)	2*i*	0.5969	0.4471	0.5147	0.066
H(24B)	2*i*	0.5491	0.3844	0.5939	0.066
H(25A)	2*i*	0.6657	0.5739	0.7748	0.065
H(25B)	2*i*	0.5700	0.6179	0.6866	0.065
H(26A)	2*i*	0.7405	0.6859	0.6267	0.056
H(26B)	2*i*	0.7809	0.7583	0.7714	0.056

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cu(1)	2*i*	0.96593(4)	0.79151(5)	0.59573(4)	0.0356(3)	0.0447(3)	0.0359(3)	0.0142(2)	0.0063(2)	0.0207(2)
O(1)	2*i*	0.3525(3)	0.8184(4)	0.1936(3)	0.050(2)	0.093(3)	0.082(2)	0.028(2)	0.008(2)	0.039(2)
O(2)	2*i*	0.3261(3)	0.6134(3)	0.0580(3)	0.059(2)	0.087(3)	0.061(2)	−0.004(2)	−0.018(2)	0.024(2)
O(3)	2*i*	0.8560(2)	0.6670(3)	0.4325(2)	0.048(2)	0.051(2)	0.042(2)	0.021(1)	0.007(1)	0.017(1)
O(4)	2*i*	1.5672(3)	0.7759(4)	1.0243(3)	0.053(2)	0.105(3)	0.079(2)	0.022(2)	0.007(2)	0.056(2)
O(5)	2*i*	1.6883(3)	0.8979(3)	0.9747(3)	0.034(2)	0.083(2)	0.093(3)	0.006(2)	0.002(2)	0.036(2)
O(6)	2*i*	1.1478(2)	0.8532(3)	0.6228(2)	0.042(2)	0.062(2)	0.055(2)	0.011(1)	0.010(1)	0.037(2)
N(1)	2*i*	0.3879(3)	0.7104(4)	0.1557(3)	0.038(2)	0.067(3)	0.055(2)	0.002(2)	0.003(2)	0.030(2)
N(2)	2*i*	0.8641(3)	0.9368(3)	0.6148(3)	0.042(2)	0.039(2)	0.031(2)	0.007(1)	0.005(1)	0.012(1)
N(3)	2*i*	1.5818(3)	0.8384(4)	0.9643(3)	0.037(2)	0.052(2)	0.058(2)	0.010(2)	0.006(2)	0.013(2)
N(4)	2*i*	0.9941(3)	0.6999(3)	0.7062(3)	0.031(2)	0.040(2)	0.038(2)	0.008(1)	0.009(1)	0.016(2)
C(1)	2*i*	0.7459(4)	0.6807(4)	0.3723(3)	0.042(2)	0.043(2)	0.035(2)	0.013(2)	0.009(2)	0.019(2)
C(2)	2*i*	0.6931(4)	0.7987(4)	0.4159(3)	0.039(2)	0.040(2)	0.036(2)	0.005(2)	0.007(2)	0.018(2)
C(3)	2*i*	0.5743(4)	0.8035(4)	0.3422(3)	0.039(2)	0.046(2)	0.044(2)	0.008(2)	0.011(2)	0.021(2)
C(4)	2*i*	0.5087(4)	0.6993(4)	0.2308(3)	0.032(2)	0.053(3)	0.039(2)	0.003(2)	0.004(2)	0.024(2)
C(5)	2*i*	0.5571(4)	0.5825(4)	0.1868(4)	0.053(3)	0.048(3)	0.038(2)	0.001(2)	0.006(2)	0.012(2)
C(6)	2*i*	0.6722(4)	0.5754(4)	0.2572(4)	0.053(3)	0.044(2)	0.042(2)	0.015(2)	0.008(2)	0.010(2)
C(7)	2*i*	0.7556(4)	0.9167(4)	0.5319(3)	0.042(2)	0.041(2)	0.047(2)	0.013(2)	0.011(2)	0.020(2)
C(8)	2*i*	0.9067(4)	1.0700(4)	0.7232(3)	0.041(2)	0.042(2)	0.038(2)	0.011(2)	0.012(2)	0.009(2)
C(9)	2*i*	0.9698(5)	1.0573(4)	0.8392(4)	0.095(4)	0.051(3)	0.039(3)	0.006(3)	0.010(2)	0.016(2)
C(10)	2*i*	1.0104(5)	1.1943(4)	0.9502(4)	0.097(4)	0.064(3)	0.037(3)	0.008(3)	0.007(3)	0.015(2)
C(11)	2*i*	1.0985(4)	1.2935(4)	0.9339(4)	0.059(3)	0.055(3)	0.056(3)	0.009(2)	−0.002(2)	0.005(2)
C(12)	2*i*	1.0361(5)	1.3034(4)	0.8152(4)	0.085(4)	0.051(3)	0.057(3)	−0.001(2)	0.014(3)	0.021(2)
C(13)	2*i*	0.9969(5)	1.1649(4)	0.7037(4)	0.080(3)	0.052(3)	0.049(3)	0.005(2)	0.019(2)	0.018(2)
C(14)	2*i*	1.2326(4)	0.7828(4)	0.7818(3)	0.037(2)	0.036(2)	0.040(2)	0.008(2)	0.007(2)	0.016(2)
C(15)	2*i*	1.2447(4)	0.8461(4)	0.7037(3)	0.039(2)	0.042(2)	0.042(2)	0.011(2)	0.008(2)	0.016(2)
C(16)	2*i*	1.3733(4)	0.9050(4)	0.7180(4)	0.047(3)	0.062(3)	0.069(3)	0.000(2)	0.013(2)	0.040(3)
C(17)	2*i*	1.4815(4)	0.9035(4)	0.8014(4)	0.039(2)	0.056(3)	0.065(3)	0.000(2)	0.011(2)	0.024(2)
C(18)	2*i*	1.4663(4)	0.8411(4)	0.8756(4)	0.031(2)	0.046(2)	0.049(2)	0.010(2)	0.005(2)	0.017(2)
C(19)	2*i*	1.3451(4)	0.7834(4)	0.8671(3)	0.042(2)	0.039(2)	0.041(2)	0.012(2)	0.008(2)	0.015(2)
C(20)	2*i*	1.1096(4)	0.7122(4)	0.7750(3)	0.042(2)	0.042(2)	0.038(2)	0.010(2)	0.010(2)	0.019(2)
C(21)	2*i*	0.8846(3)	0.6127(4)	0.7085(3)	0.040(2)	0.047(2)	0.037(2)	0.010(2)	0.009(2)	0.020(2)
C(22)	2*i*	0.8585(4)	0.4717(4)	0.6063(4)	0.045(2)	0.049(3)	0.053(3)	0.013(2)	0.018(2)	0.022(2)
C(23)	2*i*	0.7433(4)	0.3809(4)	0.6048(4)	0.055(3)	0.048(3)	0.058(3)	0.006(2)	0.014(2)	0.020(2)
C(24)	2*i*	0.6215(4)	0.4418(4)	0.5929(4)	0.040(2)	0.062(3)	0.054(3)	−0.006(2)	0.006(2)	0.025(2)
C(25)	2*i*	0.6474(4)	0.5800(4)	0.6971(4)	0.037(2)	0.070(3)	0.056(3)	0.010(2)	0.017(2)	0.027(2)
C(26)	2*i*	0.7629(3)	0.6716(4)	0.7005(4)	0.042(2)	0.047(2)	0.049(2)	0.015(2)	0.014(2)	0.018(2)

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