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N,N'-Ethylenebis(4,6-dimethoxysalicylideneiminato)copper(II) Monohydrate

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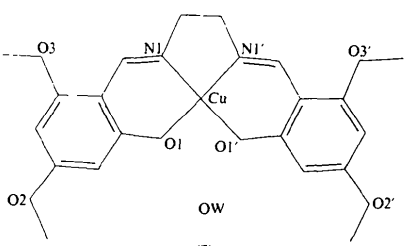
Abstract

The title complex, {2,2'-[1,2-ethanediylbis(nitrilomethylidyne)]bis(3,5-dimethoxyphenolato)-*N,N'*,-$O^1,O^{1'}$}copper(II) monohydrate, $[Cu(C_{20}H_{22}N_2O_6)].H_2O$, is mononuclear with square-planar coordination around the Cu atom. The water molecule is hydrogen bonded to a phenolic O atom of the ligand.

Comment

The synthesis of mixed complexes by electrolysis of a metal in a solution containing a weakly acidic Schiff base and an additional ligand has proved to be an efficient and high-yield route (Castro, Romero, García-Vázquez, Sousa, Castellano & Zukerman-Schpector, 1993). During an attempt to complex Cu with (2-iminomethyl)-3,5-dimethoxyphenol and ethylenediamine, following the procedure described by Habeeb, Tuck & Walter (1978), brown crystals of the title compound, (I), were obtained.



(I)

The Cu atom is coordinated by a square-planar O_2N_2 donor set; σ_{av}, defined as $[\sum_i d_i^2/(N-3)]^{1/2}$, is 0.057 Å for the least-squares plane defined by atoms N(1), N(1'), O(1), O(1') and Cu. C(10) and C(10') are displaced by −0.179 (6) and 0.302 (6) Å, respectively, from this plane. The distances and angles around the Cu atom are in good agreement with values found in other tetracoordinated copper complexes with similar ligands (Baker, Hall & Waters, 1970, and references therein). The atoms of the chelate ligand are approximately coplanar {σ_{av} = 0.088 Å for the least-squares plane through all non-H atoms of the $[Cu(C_{20}H_{22}N_2O_6)]$ molecule apart from C(10) and C(10'); the maximum deviation from this plane is that of 0.188 (4) Å for N(1')}; the ethylene bridge is in the usual *gauche* conformation, but C(10') is displaced much further from the CuO_2N_2 plane than C(10). Bond distances and angles in the ligand are in the expected range. The shortest intermolecular distance involving the Cu atom is 3.531 (4) Å to N(1') (−*x*, −*y*, 1 − *z*); this excludes the possibility of pentacoordination. The water molecule is not associated with the Cu atom but is hydrogen bonded to one of the coordinated O atoms: O(1')···O(*W*) = 2.796 (5), O(1')···H' = 2.11 Å, O(1')···H'—O(*W*) = 119°.

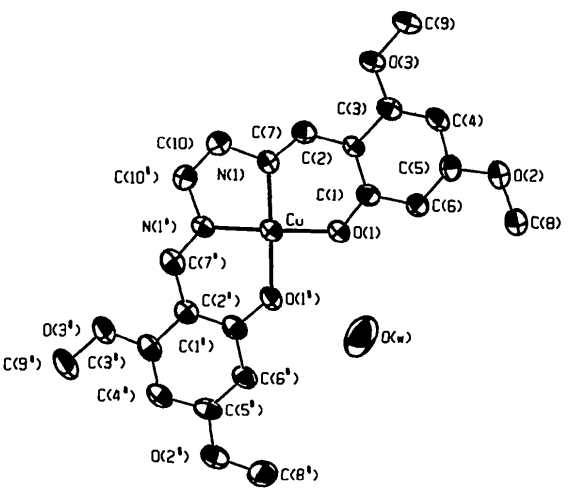


Fig. 1. A view of $[Cu(C_{20}H_{22}N_2O_6)]$ and the water molecule hydrogen bonded to O(1'). Atom labels and 50% probability ellipsoids are displayed.

Experimental

Crystal data

$[Cu(C_{20}H_{22}N_2O_6)].H_2O$
M_r = 467.96
Monoclinic
*C*2/*c*
a = 29.172 (2) Å
b = 7.7545 (9) Å
c = 18.499 (1) Å
β = 104.676 (6)°
V = 4048.2 (7) Å^3
Z = 8
D_x = 1.54 Mg m^{-3}
Cu *K*α radiation
λ = 1.5418 Å
Cell parameters from 23 reflections
θ = 16–53°
μ = 1.88 mm^{-1}
T = 293 K
Irregular
0.35 × 0.20 × 0.20 mm
Brown

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
T_{min} = 0.74, T_{max} = 1.56
2787 measured reflections
2684 independent reflections
2089 observed reflections [$I > 3\sigma(I)$]
R_{int} = 0.028
θ_{max} = 60°
$h = -32 \rightarrow 31$
$k = 0 \rightarrow 8$
$l = 0 \rightarrow 20$
2 standard reflections
frequency: 30 min
intensity variation: ±0.82%

Refinement

Refinement on F
R = 0.047
wR = 0.049
S = 1.79
2089 reflections
272 parameters
H atoms refined in fixed positions with one overall U_{iso} = 0.081(4) Å^2
$w = 1/[\sigma^2(F_o) + 0.0003F_o^2]$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
Cu	0.0142 (1)	0.2193 (1)	0.5054 (1)	3.01 (2)
O(1)	−0.0259 (1)	0.3025 (4)	0.4152 (2)	3.3 (1)
O(2)	−0.1605 (1)	0.5466 (5)	0.2482 (2)	4.6 (1)
O(3)	−0.1502 (1)	0.5447 (4)	0.5077 (2)	3.6 (1)
N(1)	−0.0263 (1)	0.3009 (5)	0.5644 (2)	3.1 (1)
C(1)	−0.0668 (1)	0.3789 (6)	0.4056 (2)	2.8 (1)
C(2)	−0.0873 (1)	0.4232 (6)	0.4651 (2)	2.6 (1)
C(3)	−0.1323 (2)	0.5094 (6)	0.4474 (3)	2.7 (1)
C(4)	−0.1555 (1)	0.5484 (6)	0.3754 (3)	3.0 (2)
C(5)	−0.1343 (2)	0.5027 (7)	0.3184 (3)	3.1 (2)
C(6)	−0.0915 (1)	0.4180 (7)	0.3313 (2)	3.0 (2)
C(7)	−0.0659 (2)	0.3832 (7)	0.5415 (3)	3.1 (2)
C(8)	−0.1415 (2)	0.5128 (9)	0.1867 (3)	5.3 (2)
C(9)	−0.1967 (2)	0.6167 (7)	0.4918 (3)	4.0 (2)
C(10)	−0.0066 (2)	0.2736 (8)	0.6456 (3)	5.0 (2)
O(1′)	0.0565 (1)	0.1380 (5)	0.4502 (2)	3.6 (1)
O(2′)	0.1941 (1)	−0.1015 (5)	0.3973 (2)	4.3 (1)
O(3′)	0.1797 (1)	−0.0854 (5)	0.6470 (2)	4.3 (1)
N(1′)	0.0512 (1)	0.1237 (5)	0.5969 (2)	3.0 (1)
C(1′)	0.0978 (1)	0.0613 (6)	0.4756 (3)	3.1 (2)
C(2′)	0.1170 (1)	0.0217 (6)	0.5518 (3)	2.7 (1)
C(3′)	0.1627 (1)	−0.0578 (7)	0.5721 (3)	3.2 (2)
C(4′)	0.1874 (1)	−0.0982 (7)	0.5206 (3)	3.3 (2)
C(5′)	0.1669 (1)	−0.0584 (7)	0.4457 (3)	3.2 (2)
C(6′)	0.1234 (1)	0.0203 (7)	0.4223 (3)	3.3 (2)
C(7′)	0.0925 (2)	0.0491 (6)	0.6087 (3)	3.2 (2)
C(8′)	0.1777 (2)	−0.0462 (8)	0.3217 (3)	5.8 (2)
C(9′)	0.2255 (2)	−0.1603 (9)	0.6713 (3)	6.0 (2)
C(10′)	0.0274 (2)	0.1292 (8)	0.6578 (3)	4.5 (2)
O(*W*)	0.0387 (2)	0.3101 (7)	0.3131 (2)	8.8 (2)

Table 2. *Selected geometric parameters* (Å, °)

Cu—O(1)	1.892 (3)	Cu—O(1′)	1.898 (3)
Cu—N(1)	1.908 (4)	Cu—N(1′)	1.912 (4)
O(1)—Cu—N(1)	93.3 (1)	N(1)—Cu—O(1′)	177.7 (2)
O(1)—Cu—O(1′)	88.8 (1)	N(1)—Cu—N(1′)	85.1 (2)
O(1)—Cu—N(1′)	175.9 (1)	O(1′)—Cu—N(1′)	92.8 (1)

Data were corrected for Lorentz and polarization effects. The structure was solved by direct methods. All H atoms were found in a difference synthesis and included as fixed contributors with an overall isotropic temperature parameter. Refinement was by full-matrix least-squares methods. Most of the calculations were performed on a VAX 6420 computer at the Instituto de Física e Química de São Carlos. Programs used were: *SHELXS*86 (Sheldrick, 1985), *SHELX*76 (Sheldrick, 1976) and *ORTEP* (Johnson, 1965).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: MU1083). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Potassium (5-Nitro-1,10-phenanthroline-N^1,N^{10})oxodiperoxovanadate(V) Dihydrate: an Insulin-Mimetic Peroxovanadate

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Abstract

The structure of the title compound, K[$VO(O_2)_2$-($C_{12}H_7N_3O_2$)].$2H_2O$, was determined. The geometry about the V atom is pentagonal bipyramidal with the pentagonal plane defined by the two peroxo groups and one N atom from the phenanthroline ligand [N(2)]. The oxo ligand lies in the plane of the