trans-Bis(3-hydroxy-2-phenyl-4H-1-benzopyran-4-onato)bis(pyridine)copper(II)

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Key indicators

Single-crystal X-ray study
$T = 296$ K
Mean σ(C–C) = 0.005 Å
R factor = 0.043
wR factor = 0.152
Data-to-parameter ratio = 16.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title complex, $[Cu(C_{15}H_9O_3)_2(C_5H_5N)_2]$, has a distorted octahedral coordination in which the Cu^{II} atom lies on a center of symmetry. The bidentate ligands lie in *trans* positions with respect to one another, forming the equatorial plane, with two pyridyl ligands occupying the axial positions.

Comment

Flavonol (3-hydroxyflavone) is the model compound of quercetin (3′-4′,5,7-tetrahydroxyflavonol), which is a component of rutin and which is oxygenated to the corresponding phenolic carboxylic acid ester catalysed by the copper-containing quercetin 2,3-dioxygenase (Fusetti *et al.*, 2002). The copper complex of flavonol also degraded to the corresponding depside, catalysed by Cu^I and Cu^{II} flavonolate complexes (Balogh-Hergovich *et al.*, 1991). The molecular structures of a Cu^I complex (Speier *et al.*, 1990) and a Cu^{II} complex (Balogh-Hergovich *et al.*, 1991) of flavonol have been determined to understand the coordination modes. In the case of the Cu^I complex, one flavonol ligand coordinates to the central Cu^I by its 3-hydroxy and 4-carbonyl groups, together with two triphenylphosphines, to form a distorted tetrahedron (Speier *et al.*, 1990). In the case of the Cu^{II} complex, two flavonolate ligands are coordinated to Cu^{II} by their 3-hydroxy and 4-carbonyl groups to form a square-planar geometry (Balogh-Hergovich *et al.*, 1991). In the modeling of the substrate into the active site of the copper-containing quercetin 2,3-dioxygenase from *Aspergillus japonicus*, the Cu atom is surrounded by six coordinated atoms (Fusetti *et al.*, 2002) and the 3-hydroxy and 4-carbonyl groups of the quercetin molecule are ligated to the Cu atom. In this study, we report the structure of a copper complex of flavonol, (I).

(I)
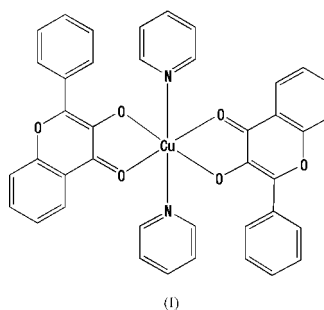
The molecular structure of (I) is shown in Fig. 1. The Cu^{II} atom has a distorted octahedral coordination geometry, in the *trans* form, defined by two O atoms of the 3-hydroxy and 4-carbonyl groups of two bidentate ligands in the equatorial plane, and two axial N atoms of two pyridyl ligands. The Cu

atom lies on a center of symmetry. The geometric parameters are listed in Table 1. The 3-hydroxyl group is deprotonated, and the C1—O1 bond is shorter than the other C—O single bonds. The short C2—O2 bond distance reflects the ketonic form of the 4-carbonyl group. The coordination bond distance in the axial direction, Cu1—N1, is longer than those in the equatorial plane. This is commonly observed in the distorted octahedral coordination geometry of the Cu^{II} complexes and is explained by a Jahn–Teller effect. The flavonol moiety is almost planar [C1—C9—C10—C15 = $-8.0\,(5)°$], and atoms Cu1, O1, C1, C2 and O2 form a five-membered ring.

Experimental

Yellow prismatic crystals of (I) were obtained by slow evaporation of a mixture of flavonol and $Cu(OH)_2$ in pyridine solution (molar ratio 2:1).

Crystal data

[Cu(C15H9O3)2(C5H5N)2]
$M_r = 696.19$
Triclinic, $P\bar{1}$
$a = 8.539\,(2)$ Å
$b = 10.029\,(2)$ Å
$c = 11.098\,(2)$ Å
$\alpha = 114.70\,(1)°$
$\beta = 97.18\,(2)°$
$\gamma = 107.00\,(1)°$
$V = 791.6\,(3)$ Å^3

$Z = 1$
$D_x = 1.460$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25
    reflections
$\theta = 13.9–14.9°$
$\mu = 0.74$ mm^{-1}
$T = 296.2$ K
Prism, yellow
$0.30 \times 0.30 \times 0.20$ mm

Data collection

Rigaku AFC-5R diffractometer
ω–2θ scans
Absorption correction: ψ scan
    (North et al., 1968)
    $T_{min} = 0.799$, $T_{max} = 0.859$
3834 measured reflections
3632 independent reflections
2303 reflections with $I > 2\sigma(I)$

$R_{int} = 0.018$
$\theta_{max} = 27.5°$
$h = -11 \rightarrow 10$
$k = 0 \rightarrow 13$
$l = -14 \rightarrow 13$
3 standard reflections
    every 150 reflections
    intensity decay: 0.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.152$
$S = 0.95$
3632 reflections
223 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$
    where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.001$
$\Delta\rho_{max} = 0.56$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

|			
Cu1—O1	1.931 (2)	Cu1—N1	2.495 (4)
Cu1—O1^i	1.931 (2)	Cu1—N1^i	2.495 (4)
Cu1—O2	2.039 (2)	O1—C1	1.312 (3)
Cu1—O2^i	2.039 (2)	O2—C2	1.255 (3)
O1—Cu1—O1^i	180.0	O2—Cu1—N1	90.1 (1)
O1—Cu1—O2	83.29 (9)	O2—Cu1—N1^i	89.9 (1)
O1—Cu1—O2^i	96.71 (9)	O2^i—Cu1—N1	89.9 (1)
O1—Cu1—N1	91.3 (1)	O2^i—Cu1—N1^i	90.1 (1)
O1—Cu1—N1^i	88.7 (1)	N1—Cu1—N1^i	180.0
O1^i—Cu1—O2	96.71 (9)	Cu1—O1—C1	112.0 (2)
O1^i—Cu1—O2^i	83.29 (9)	Cu1—O2—C2	109.4 (2)
O1^i—Cu1—N1	88.7 (1)	Cu1—N1—C16	120.9 (3)
O1^i—Cu1—N1^i	91.3 (1)	Cu1—N1—C20	120.5 (3)
O2—Cu1—O2^i	180.0		

Symmetry code: (i) $-x, -y, 1-z$.
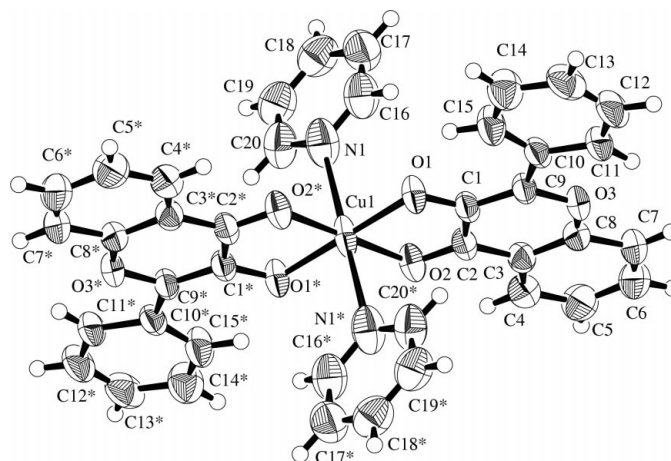
Figure 1
ORTEPII (Johnson, 1976) drawing of (I), with the atomic numbering scheme. Ellipsoids for non-H atoms are drawn at the 50% probability level. Atoms marked with an asterisk are at the symmetry position ($-x$, $-y$, $1-z$).

After their location in a difference map, all H atoms were placed at calculated positions [C—H = 0.93 Å and $U_{iso}(H) = 1.5U_{eq}(C)$] and refined as riding.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation and Rigaku, 2000); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation and Rigaku, 2000); program(s) used to solve structure: *SIR*97 (Altomare et al., 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

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