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Bis(flufenamato-κO)bis(3-pyridylmethanol-κ^2N,O)copper(II)

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The crystal structure of the title complex, $[Cu(C_{14}H_9NF_3O_2)_2(C_6H_6NO)_2]_n$, has been studied by single-crystal X-ray diffraction at 100 (2) K. The polymeric structure is centrosymmetric and forms two-dimensional sheets. The Cu^{II} centre has octahedral coordination. It is bonded in a *trans*-square-planar arrangement to the N atoms of two ronicol molecules and one carboxylate O atom from each of two flufenamate anions. The axial positions are also occupied by 3-pyridylmethanol but coordinated through oxygen rather than the pyridyl N atom.

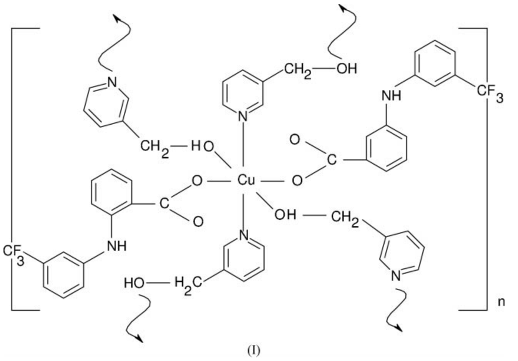
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.039
wR factor = 0.095
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.

Comment

The synthesis and structural characterization of a copper(II) complex with a potent non-steroidal anti-inflammatory drug, flufenamic acid (fluf), with formula $[Cu(fluf)_2(ron)_2]$ (ron is ronicol; 3-pyridylmethanol) has been investigated. The complex is shown in Fig. 1. Selected bond distances and angles are summarized in Table 1. The asymmetric unit consists of a Cu atom located on an inversion centre, one flufenamate anion and one 3-pyridylmethanol molecule. The coordination sphere of the Cu atom consists of two O atoms from flufenamate [Cu1—O1 = 1.974 (1) Å] and its symmetry equivalent, two N atoms from 3-pyridylmethanol [Cu1—N1 = 2.028 (2) Å] and its symmetry equivalent and, in the axial sites also, from symmetry-equivalent 3-pyridylmethanol species but O-bound rather than N-bound [Cu1—O3b = 2.404 (1) Å; symmetry position of the ronicol: $x, \frac{1}{2} - y, z - \frac{1}{2}$ and $-x, y - \frac{1}{2}, \frac{1}{2} - z$].



(I)

The intramolecular hydrogen-bonding interaction [N2—H2N···O2, with an O···N distance of 2.645 (2) Å] stabilizes the structure (Table 2). The crystal structure of the title complex consists of two-dimensional sheets. The layers lie parallel to the ab plane. The crystal structure of the compound under study can be compared with those of $[CuX_2(ron)_2]_n$ where X = salicylate (sal) (Hoang *et al.*, 1992), niflumate (nif) (Valach *et al.*, 1997) and clofibriate (clof) (Moncoľ *et al.*, 2001). The Cu—O$_{eq}$ (the O atom of the carboxylic acid group), Cu—N$_{eq}$ (the N atom of the pyridine ring of ronicol) and Cu—O$_{ax}$

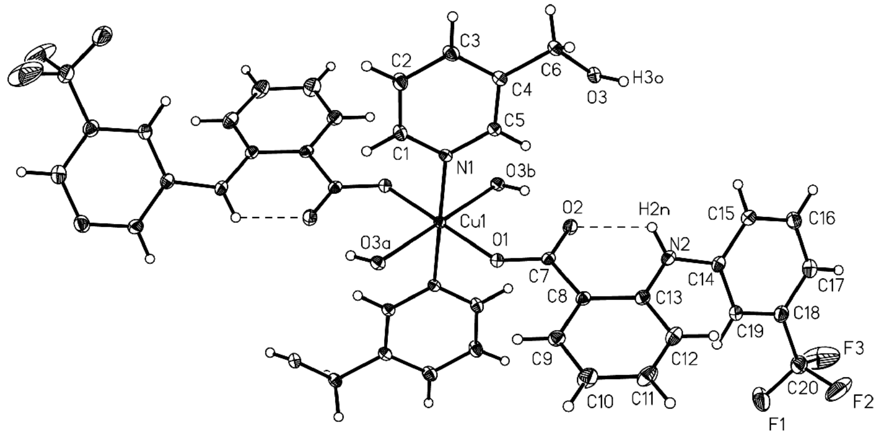


Figure 1
The structure of the title compound. Displacement ellipsoids are drawn at the 40% probability level and H atoms are shown as spheres of arbitrary radii. Minor disorder-component atoms F11, F21 and F31 have been omitted for clarity.

(the O atom of the hydroxyl group of ronicol) lengths are: 1.944 (4), 2.039 and 2.622 (4) Å for salicylate, 1.946 (3), 2.038 (3) and 2.573 (3) Å for niflumate, and 1.989 (2), 2.008 (2) and 2.441 (1) Å for clofibriate. In the present crystal structure, the equivalent bond lengths are comparable (Table 1). All four compounds have polymeric structures with axially elongated octahedral geometry about each Cu^{II} atom of the CuO_4N_2 chromophore, as predicted by the Jahn–Teller effect.

Experimental

The title complex was prepared by addition of 3-pyridylmethanol (0.02 mol) to copper(II) flufenamate (0.01 mol) in hot methanol. The mixture was stirred, filtered and left to cool and stand at room temperature. Green air-stable crystals were collected. Analysis found: C 56,96, H,3.75, N,6.78, Cu 7.22%; calculated for $[Cu(fluf)_2(ron)_2]$: C 57.05, H 3.83, N,7.54, Cu 7.54%.

Crystal data

$[Cu(C_{14}H_9NF_3O_2)_2(C_6H_6NO)_2]$
$M_r = 842.25$
Monoclinic, $P2_1/c$
$a = 18.787$ (4) Å
$b = 10.576$ (2) Å
$c = 9.097$ (2) Å
$\beta = 95.51$ (3)°
$V = 1799.1$ (7) Å^3
$Z = 2$

$D_x = 1.555$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 4.5$–12.5°
$\mu = 0.69$ mm^{-1}
$T = 293$ (2) K
Prism, green
0.20 × 0.20 × 0.15 mm

Data collection

Kuma KM4CCD diffractometer
ω scans
Absorption correction: refined from ΔF (*COSABS*99; Starynowicz, 1999)
$T_{min} = 0.870$, $T_{max} = 0.901$
11922 measured reflections

4474 independent reflections
3498 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 28.6°$
$h = -24 \rightarrow 25$
$k = -14 \rightarrow 14$
$l = -12 \rightarrow 6$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.095$
$S = 1.10$
4474 reflections
274 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0549P)^2 + 0.5478P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—O1	1.9747 (14)	Cu1—O3^{i}	2.404 (1)
Cu1—N1	2.0279 (16)		
O1—Cu1—N1	91.33 (6)	O1^{iii}—Cu1—O3^{ii}	84.43 (5)
O1—Cu1—O3^{ii}	95.57 (5)	N1—Cu1—O3^{i}	88.77 (5)

Symmetry codes: (i) $-x, y-\frac{1}{2}, \frac{1}{2}-z$; (ii) $x, \frac{1}{2}-y, z-\frac{1}{2}$; (iii) $-x, -y, -z$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2N···O2	0.86	2.12	2.645 (2)	119

All the H atoms were placed in geometrically calculated positions, except for the alkoxyl H atom, which was located in a difference Fourier map. The H atoms were not refined and the U_{iso} values were set at 0.05 Å^2, except for H3O which has been refined with the O3—H3O distance restrained to 0.84 (1) Å. The F atoms of the CF_3 group are disordered, occupying two statistical positions, represented by F1/F2/F3 and F11/F21/F31, with occupancy factors of 0.919 (4) and 0.081 (4), respectively. Atoms F1, F2 and F3 were refined anisotropically and atoms F11, F21 and F31 were refined isotropically. .

Data collection: *CRYSALIS* (Kuma, 1998); cell refinement: *CRYSALIS*; data reduction: *CRYSALIS*; program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP* (Johnson, 1965); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

References

Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Hoang, N. N., Valach, F., Macašková, Ľ. & Melník, M. (1992). *Acta Cryst.* C**48**, 1933–1935.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Kuma (1998). *KM4CCD System Software*. Version 1.149. Kuma Diffraction Instruments, Wrocław, Poland, and Université Lausanne, Switzerland.
Moncol, J., Koman, M., Melnik, M. & Glowiak, T. (2001). *CrystEngComm*, article 54, 1-3.

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Sheldrick, G. M. (1985). *SHELXS*86. In *Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford University Press.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Starynowicz, P. (1999). *COSABS*99. University of Wrocław, Poland.

Valach, F., Tokarčík, M., Kubinec, P., Melník, M. & Macašková, Ľ. (1997). *Polyhedron*, **16**, 1461–1465.

m592 Štefan Lörinc *et al.* • $[Cu(C_{14}H_9NF_3O_2)_2(C_6H_6NO)_2]$ *Acta Cryst.* (2004). E**60**, m590–m592