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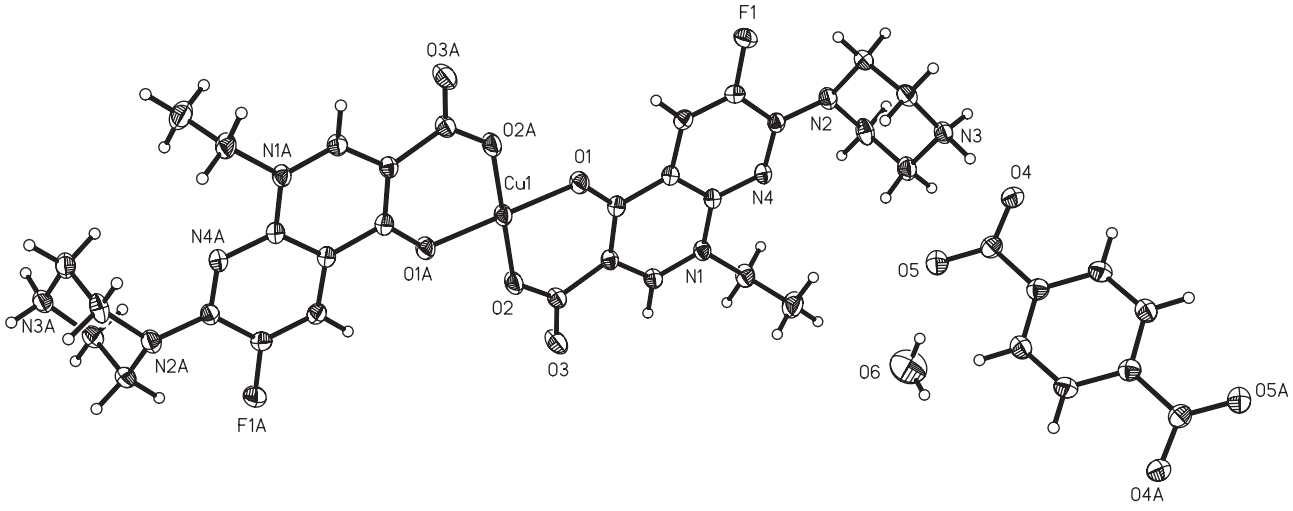
Crystal structure of bis(1-ethyl-6-fluoro-4-oxido-7-(piperazin-1-ium-1-yl)-1,8-naphthyridin-1-ium-3-carboxylate-κ^2*O*,*O*′)copper(II) benzene-1,4-dicarboxylate dihydrate, $C_{38}H_{42}F_2CuN_8O_{12}$



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Abstract

$C_{38}H_{42}F_2CuN_8O_{12}$, triclinic, $P\bar{1}$ (no. 2), $a = 5.4709(2)$ Å, $b = 10.4100(4)$ Å, $c = 17.3373(6)$ Å, $\alpha = 95.014(2)°$, $\beta = 92.041(2)°$, $\gamma = 103.223(2)°$, $V = 955.96(6)$ Å^3, $Z = 1$, $R_{gt}(F) = 0.0347$, $wR_{ref}(F^2) = 0.1125$, T = 296 K.

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Table 1: Data collection and handling.

Crystal:	Green blocks Size 0.25 × 0.25 × 0.22 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	6.6 cm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω
$2\theta_{max}$, completeness:	50°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13821, 3380, 0.031
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3071
$N(param)_{refined}$:	292
Programs:	SHELX [5], Bruker programs [6]

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The crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

A mixture of $Cu(CH_3COO)_2{\cdot}H_2O$ (0.25 mmol), enoxacin (0.50 mmol), 1,4-benzenedicarboxylic acid (0.25 mmol), sodium hydroxide (0.5 mmol) and water (15 mL) was

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1517(4)	1.0768(2)	0.84813(14)	0.0362(5)
C2	0.3125(4)	0.9770(2)	0.84485(13)	0.0315(5)
C3	0.4393(4)	0.9653(2)	0.77824(14)	0.0342(5)
H3	0.4188	1.0190	0.7395	0.041*
C4	0.3374(4)	0.8959(2)	0.90469(13)	0.0314(5)
C5	0.5027(4)	0.8074(2)	0.89095(13)	0.0304(5)
C6	0.6247(4)	0.7999(2)	0.82163(13)	0.0303(5)
C7	0.5511(4)	0.7232(2)	0.94613(13)	0.0354(5)
H7	0.4786	0.7246	0.9938	0.043*
C8	0.7025(5)	0.6411(2)	0.92933(14)	0.0369(5)
C9	0.8122(4)	0.6343(2)	0.85609(13)	0.0321(5)
C10	0.7108(5)	0.8736(3)	0.69031(14)	0.0438(6)
H10A	0.8824	0.8657	0.6996	0.053*
H10B	0.7165	0.9548	0.6662	0.053*
C11	0.5699(7)	0.7572(3)	0.63610(17)	0.0629(8)
H11A	0.3988	0.7636	0.6279	0.094*
H11B	0.5730	0.6764	0.6585	0.094*
H11C	0.6482	0.7574	0.5874	0.094*
C12	1.0663(5)	0.5532(3)	0.75942(17)	0.0455(6)
H12A	1.2304	0.5326	0.7617	0.055*
H12B	1.0865	0.6412	0.7425	0.055*
C13	0.8934(5)	0.4542(3)	0.70188(16)	0.0455(6)
H13A	0.7344	0.4795	0.6958	0.055*
H13B	0.9674	0.4537	0.6519	0.055*
C14	0.7607(5)	0.3167(2)	0.80788(15)	0.0409(6)
H14A	0.7506	0.2294	0.8249	0.049*
H14B	0.5936	0.3337	0.8080	0.049*
C15	0.9363(5)	0.4192(2)	0.86311(15)	0.0446(6)
H15A	0.8698	0.4191	0.9142	0.053*
H15B	1.0988	0.3970	0.8670	0.053*
C16	−0.0540(5)	0.1211(3)	0.52077(15)	0.0429(6)
H16	−0.0922	0.2024	0.5344	0.051*
C17	0.1556(4)	0.0913(2)	0.55527(14)	0.0372(5)
C18	0.2066(5)	−0.0309(3)	0.53370(15)	0.0433(6)
H18	0.3466	−0.0524	0.5564	0.052*
C19	0.3276(5)	0.1882(2)	0.61431(14)	0.0393(6)
Cu1	0.0000	1.0000	1.0000	0.03519(16)
F1	0.7598(3)	0.56743(17)	0.98475(9)	0.0575(4)
H3A	0.982(6)	0.292(3)	0.7225(16)	0.044(8)*
H6A	0.249(6)	0.417(3)	0.5512(15)	0.065*
H6B	0.124(3)	0.434(2)	0.4914(14)	0.065*
H3B	0.717(6)	0.254(3)	0.693(2)	0.069(10)*
N1	0.5906(4)	0.88150(18)	0.76559(11)	0.0333(4)
N2	0.9676(4)	0.5520(2)	0.83669(12)	0.0391(5)
N3	0.8490(4)	0.3195(2)	0.72848(13)	0.0412(5)
N4	0.7717(3)	0.71567(18)	0.80462(11)	0.0329(4)
O1	0.2272(3)	0.89333(17)	0.96832(10)	0.0406(4)
O2	0.0266(3)	1.08942(18)	0.90826(10)	0.0451(4)
O3	0.1469(4)	1.1425(2)	0.79313(12)	0.0570(5)
O4	0.5154(3)	0.15093(18)	0.64053(10)	0.0447(4)
O5	0.2783(4)	0.29689(19)	0.63427(11)	0.0518(5)
O6	0.2458(6)	0.4757(3)	0.5212(2)	0.0999(9)

stirred for 20 min in air. The mixture was then transferred to a 25 mL Teflon reactor and kept at 448 K for 72 h under autogenous pressure. After cooling, green single crystals of title compound were obtained from the reaction mixture.

Experimental details

The H on Nitrogen were located in difference Fourier maps and refined in the riding model approximation, with N—H and distances restrained (0.86(1) or 0.85(1) Å) and $U(H) = 1.5U_{eq}(N)$.

Discussion

Enoxacin (Henox), $C_{15}H_{17}F_2N_4O_3$ (systematic name 1-ethyl-6-fluoro-1,4-dihydro-4-oxo-7-(1-piperazinyl)-1,8-naphtyridine-3-carboxylic acid) is a member of the fluoroquinolone (flqu) family of antibiotics, widely used in veterinary clinical practice because of its wide antibiotic spectrum and its excellent bactericidal activity [1]. Our interest in enoxacin and related fluoroquinolones is focused on their potential as multidentate/bridging ligands in coordination chemistry. The manganese, cobalt and zinc complexe derivatives of enoxacin have been reported [2–4]. In the title compound the Cu^{II} atom exhibits a CuO4 square geometry defined by two neutral bidentate O,O-coordinated 1-ethyl-6-fluoro-1,4-dihydro-4-oxo-7-(1-piperazinyl)-1,8-naphtyridine-3-carboxylate (Henox) zwitterionic ligands. The charge-balancing benzene-1,4-dicarboxylate (1,4-bdc) dianion is also centrosymmetric.

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