Crystal structure of aqua-*bis*-[1-cyclopropyl-7-(4-ethylpiperazin-1-yl)-6-fluor-4-oxo-1,4-dihydrochinolin-3-carboxylato]copper(II), $[Cu(C_{19}H_{21}N_3FO_3)_2(H_2O)]\cdot 2.5(H_2O)$, $C_{38}H_{49}CuF_2N_6O_{9.50}$

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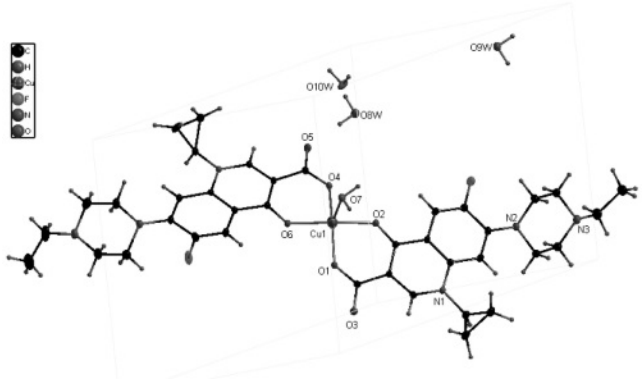

Abstract

$C_{38}H_{49}CuF_2N_6O_{9.50}$, triclinic, $P\bar{1}$ (no. 2), a = 11.891(2) Å, b = 12.354(3) Å, c = 15.369(3) Å, α = 69.25(3)°, β = 70.34(3)°, γ = 84.00(3)°, V = 1987.9 Å^3, Z = 2, $R_{gt(F)}$ = 0.0437, $wR_{ref(F^2)}$ = 0.1508, T = 293 K.

Table 1. Data collection and handling.

Crystal:	blue blocks, size 0.20×0.21×0.23 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	6.21 cm^{-1}
Diffractometer, scan mode:	Bruker P4, ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	15763, 6983
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5177
$N(param)_{refined}$:	530
Programs:	SHELX [4]

Source of material

A mixture of $Cu(CH_3COO)_2\cdot H_2O$ (0.25 mmol), enrofloxacin (0.5 mmol), sodium hydroxide (1 mmol), and water (15 mL) was stirred for 30 min in air. The mixture was then transferred to a 25 mL Teflon-lined autoclave, which was heated to 445K for 48 h under autogenous pressure. After cooling, the bomb was opened and blue blocks of the title compound were recovered from the reaction mixture by vacuum filtration and rinsing with water. Crystals suitable for single crystal X-ray diffraction were selected directly from the sample as prepared.

Discussion

Enrofloxacin (Henro), $C_{19}H_{22}FN_3O_3$ (systematic name: 1-cyclopropyl-7-(4-ethylpiperazin-1-yl)-6-fluor-4-oxo-1,4-dihydrochinolin-3-carboxylic acid), is a member of the fluoroquinolone (flqu) family of antibiotics [1], widely used in veterinary clinical practice because of its wide antibiotic spectrum and its excellent bactericidal activity [2]. Our interest in enrofloxacin and related fluoroquinolones is focused on their potential as multidentate/bridging ligands in coordination chemistry. The cadmium and lead complexe of enrofloxacin anion have been reported [3]. In the title compound, $[Cu(C_{19}H_{21}N_3FO_3)_2(H_2O)]\cdot 2.5(H_2O)$, the Cu^{II} atom exhibits a distorted $[CuO_5]$ square-pyramidal geometry defined by two bidentate *O*,*O*-bonded enro anions in the basal plane and one water molecule in the apical position. A network of O–H···O and O–H···N hydrogen bonds is formed between the Cu^{II} complexes and the uncoordinated water molecules.

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(3)	2*i*		0.3800	0.6588	–0.1725	0.050
H(4)	2*i*		0.3707	1.0314	0.2050	0.055
H(5)	2*i*		0.6997	0.4102	–0.1923	0.043
H(10A)	2*i*		–0.0954	1.2795	0.1770	0.070
H(10B)	2*i*		–0.2072	1.2607	0.2787	0.070
H(12)	2*i*		0.7447	0.6042	–0.0062	0.043
H(13)	2*i*		–0.0627	0.9864	0.1504	0.046
H(19)	2*i*		–0.0029	1.1650	0.3418	0.048
H(20A)	2*i*		0.4084	0.3777	–0.2885	0.086
H(20B)	2*i*		0.4676	0.2612	–0.2426	0.086
H(24A)	2*i*		0.2741	1.2256	0.5262	0.061
H(24B)	2*i*		0.2863	1.3611	0.4819	0.061
H(25A)	2*i*		–0.0146	1.1400	0.4972	0.062
H(25B)	2*i*		–0.0024	1.2757	0.4521	0.062
H(26A)	2*i*		0.1993	1.3651	0.3653	0.061
H(26B)	2*i*		0.3106	1.2840	0.3572	0.061
H(27A)	2*i*		–0.0350	1.2158	0.6211	0.064
H(27B)	2*i*		0.0778	1.1373	0.6098	0.064
H(28A)	2*i*		0.4431	0.2661	–0.3906	0.085
H(28B)	2*i*		0.5228	0.3792	–0.4483	0.085
H(29A)	2*i*		0.6737	0.2581	–0.2562	0.073
H(29B)	2*i*		0.7522	0.3712	–0.3191	0.073
H(30A)	2*i*		–0.2967	1.1240	0.2426	0.071
H(30B)	2*i*		–0.1849	1.1429	0.1408	0.071
H(31A)	2*i*		0.7340	0.3646	–0.4624	0.108
H(31B)	2*i*		0.7810	0.2446	–0.4090	0.108
H(33A)	2*i*		0.9027	0.5402	–0.2400	0.082
H(33B)	2*i*		0.9416	0.4057	–0.1980	0.082
H(34A)	2*i*		0.9681	0.4408	–0.0626	0.072
H(34B)	2*i*		0.9292	0.5753	–0.1046	0.072
H(35A)	2*i*		0.1701	1.2371	0.6813	0.080
H(35B)	2*i*		0.0438	1.2929	0.7030	0.080
H(36A)	2*i*		0.2609	1.4181	0.6094	0.121
H(36B)	2*i*		0.1748	1.4064	0.7152	0.121
H(36C)	2*i*		0.1360	1.4775	0.6232	0.121
H(37A)	2*i*		0.6629	0.2693	–0.5480	0.132
H(37B)	2*i*		0.7175	0.1566	–0.4922	0.132
H(38A)	2*i*		0.5341	0.0683	–0.4356	0.223
H(38B)	2*i*		0.5961	0.1054	–0.5496	0.223
H(38C)	2*i*		0.4925	0.1796	–0.5055	0.223

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Table 2. continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(1D)	2*i*		0.778(4)	0.384(4)	–0.049(3)	0.04(1)
H(2D)	2*i*		–0.163(5)	1.057(4)	0.333(4)	0.07(2)
H(1W)	2*i*		0.740(4)	0.817(4)	0.134(4)	0.080
H(2W)	2*i*		0.836(1)	0.846(5)	0.137(4)	0.080
H(3W)	2*i*		0.8584	0.7417	0.9973	0.151
H(4W)	2*i*		0.9545	0.7641	1.0051	0.151
H(6W)	2*i*		0.4493	1.0567	–0.0987	0.151
H(5W)	2*i*		0.3718	1.0459	–0.1343	0.151
H(8W)	2*i*	0.5	0.9582	0.5443	0.5192	0.151
H(7W)	2*i*	0.5	1.0118	0.4526	0.5471	0.151

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

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	2*i*		0.5987(3)	0.7562(4)	0.0319(3)	0.034(2)	0.046(2)	0.050(3)	0.005(2)	–0.016(2)	–0.031(2)
C(2)	2*i*		0.5061(3)	0.6850(3)	–0.0684(3)	0.031(2)	0.032(2)	0.037(2)	0.003(2)	–0.012(2)	–0.017(2)
C(3)	2*i*		0.4457(4)	0.6106(4)	–0.1770(3)	0.041(2)	0.044(2)	0.056(3)	0.016(2)	–0.027(2)	–0.030(2)
C(4)	2*i*		0.2951(4)	1.0565(4)	0.2329(3)	0.034(2)	0.064(3)	0.056(3)	0.012(2)	–0.017(2)	–0.041(2)
C(5)	2*i*		0.6367(3)	0.4616(3)	–0.1901(3)	0.037(2)	0.038(2)	0.045(2)	0.006(2)	–0.016(2)	–0.027(2)
C(6)	2*i*		0.5244(3)	0.6109(3)	–0.1270(3)	0.032(2)	0.036(2)	0.045(2)	0.006(2)	–0.016(2)	–0.025(2)
C(7)	2*i*		0.5959(3)	0.6824(3)	–0.0257(3)	0.029(2)	0.038(2)	0.044(2)	0.004(2)	–0.013(2)	–0.025(2)
C(8)	2*i*		0.1066(3)	0.8767(4)	0.0664(3)	0.031(2)	0.046(2)	0.056(3)	0.004(2)	–0.018(2)	–0.030(2)
C(9)	2*i*		0.5592(3)	0.4635(3)	–0.2406(3)	0.040(2)	0.038(2)	0.038(2)	0.005(2)	–0.012(2)	–0.022(2)
C(10)	2*i*		–0.1568(4)	1.2276(4)	0.2304(4)	0.050(3)	0.062(3)	0.073(3)	0.019(2)	–0.021(2)	–0.040(3)
C(11)	2*i*		0.6224(3)	0.5356(3)	–0.1358(3)	0.027(2)	0.033(2)	0.038(2)	–0.002(2)	–0.009(2)	–0.018(2)
C(12)	2*i*		0.6887(3)	0.6065(3)	–0.0369(3)	0.029(2)	0.041(2)	0.047(2)	0.002(2)	–0.015(2)	–0.025(2)
C(13)	2*i*		0.0039(3)	0.9984(3)	0.1648(3)	0.032(2)	0.044(2)	0.051(3)	0.004(2)	–0.018(2)	–0.026(2)
C(14)	2*i*		0.2135(3)	0.9710(3)	0.1426(3)	0.031(2)	0.039(2)	0.039(2)	0.004(2)	–0.011(2)	–0.021(2)
C(15)	2*i*		0.1093(3)	0.9503(3)	0.1249(3)	0.029(2)	0.036(2)	0.041(2)	0.007(2)	–0.013(2)	–0.023(2)
C(16)	2*i*		0.0861(3)	1.0793(3)	0.2485(3)	0.034(2)	0.039(2)	0.038(2)	0.004(2)	–0.012(2)	–0.021(2)
C(17)	2*i*		0.1663(4)	1.1518(4)	0.3439(3)	0.042(2)	0.050(2)	0.042(2)	0.005(2)	–0.012(2)	–0.030(2)
C(18)	2*i*		0.1980(3)	1.0369(3)	0.2072(3)	0.032(2)	0.040(2)	0.041(2)	0.008(2)	–0.015(2)	–0.025(2)
C(19)	2*i*		0.0717(4)	1.1367(4)	0.3157(3)	0.035(2)	0.051(2)	0.043(2)	0.009(2)	–0.013(2)	–0.029(2)
C(20)	2*i*		0.4809(5)	0.3332(5)	–0.2979(5)	0.056(3)	0.092(4)	0.095(4)	0.002(3)	–0.025(3)	–0.064(4)
C(21)	2*i*		0.4632(4)	0.5419(4)	–0.2317(3)	0.053(3)	0.049(2)	0.059(3)	0.012(2)	–0.037(2)	–0.032(2)
C(22)	2*i*		–0.1287(4)	1.1030(4)	0.2655(4)	0.033(2)	0.059(3)	0.061(3)	0.011(2)	–0.017(2)	–0.037(3)
C(23)	2*i*		0.2790(4)	1.1113(4)	0.2978(3)	0.035(2)	0.068(3)	0.059(3)	0.005(2)	–0.020(2)	–0.042(3)
C(24)	2*i*		0.2371(4)	1.2957(4)	0.4950(3)	0.046(2)	0.065(3)	0.059(3)	0.006(2)	–0.019(2)	–0.040(3)
C(25)	2*i*		0.0346(4)	1.2057(4)	0.4834(3)	0.038(2)	0.072(3)	0.056(3)	–0.004(2)	–0.006(2)	–0.041(3)
C(26)	2*i*		0.2310(4)	1.2928(4)	0.3991(3)	0.051(3)	0.061(3)	0.047(3)	–0.008(2)	–0.007(2)	–0.033(2)
C(27)	2*i*		0.0443(4)	1.2093(4)	0.5776(3)	0.051(3)	0.064(3)	0.048(3)	–0.003(2)	–0.006(2)	–0.030(2)
C(28)	2*i*		0.5098(5)	0.3072(5)	–0.3930(4)	0.070(3)	0.096(4)	0.090(4)	0.018(3)	–0.043(3)	–0.070(4)
C(29)	2*i*		0.6853(4)	0.3277(5)	–0.3135(4)	0.061(3)	0.076(3)	0.078(4)	0.021(3)	–0.035(3)	–0.057(3)
C(30)	2*i*		–0.2125(4)	1.1426(4)	0.2079(4)	0.039(2)	0.083(4)	0.078(4)	0.025(2)	–0.027(2)	–0.052(3)
C(31)	2*i*		0.7126(5)	0.2950(7)	–0.4048(5)	0.069(4)	0.126(6)	0.108(5)	0.018(4)	–0.021(4)	–0.090(5)
C(32)	2*i*		0.8037(4)	0.4557(4)	–0.0907(3)	0.039(2)	0.036(2)	0.055(3)	0.008(2)	–0.020(2)	–0.027(2)
C(33)	2*i*		0.9050(4)	0.4737(5)	–0.1832(4)	0.039(3)	0.101(4)	0.076(4)	0.024(3)	–0.016(2)	–0.053(3)
C(34)	2*i*		0.9214(4)	0.4955(4)	–0.0990(4)	0.038(2)	0.065(3)	0.105(4)	0.019(2)	–0.034(3)	–0.057(3)
C(35)	2*i*		0.1240(5)	1.3032(5)	0.6564(4)	0.081(4)	0.084(4)	0.049(3)	0.001(3)	–0.022(3)	–0.038(3)
C(36)	2*i*		0.1789(5)	1.4111(6)	0.6505(5)	0.072(4)	0.115(5)	0.084(4)	–0.005(3)	–0.021(3)	–0.071(4)
C(37)	2*i*		0.6446(7)	0.2006(7)	–0.4895(6)	0.135(6)	0.138(6)	0.123(6)	0.046(5)	–0.068(5)	–0.109(6)
C(38)	2*i*		0.5600(8)	0.1330(9)	–0.4955(8)	0.149(8)	0.183(9)	0.22(1)	0.043(7)	–0.096(8)	–0.163(9)
Cu(1)	2*i*		0.36543(4)	0.84828(4)	0.01849(4)	0.0298(3)	0.0395(3)	0.0478(3)	0.0058(2)	–0.0148(2)	–0.0299(2)
F(1)	2*i*		0.3875(3)	0.5515(3)	–0.2831(3)	0.100(2)	0.096(2)	0.125(3)	0.056(2)	–0.090(2)	–0.085(2)
F(2)	2*i*		0.3733(2)	1.1237(3)	0.3245(2)	0.045(2)	0.118(2)	0.101(2)	0.020(2)	–0.036(2)	–0.086(2)
N(1)	2*i*		–0.0104(3)	1.0613(3)	0.2228(2)	0.029(2)	0.043(2)	0.046(2)	0.008(1)	–0.014(1)	–0.027(2)
N(2)	2*i*		0.1541(3)	1.1960(3)	0.4184(3)	0.041(2)	0.065(2)	0.049(2)	–0.003(2)	–0.008(2)	–0.040(2)
N(3)	2*i*		0.1183(3)	1.3052(3)	0.5615(3)	0.050(2)	0.053(2)	0.040(2)	0.006(2)	–0.017(2)	–0.027(2)
N(4)	2*i*		0.5778(3)	0.3975(3)	–0.3015(3)	0.043(2)	0.052(2)	0.052(2)	0.009(2)	–0.020(2)	–0.036(2)
N(5)	2*i*		0.6150(4)	0.2381(4)	–0.4057(3)	0.059(2)	0.074(3)	0.079(3)	0.009(2)	–0.024(2)	–0.057(3)
N(6)	2*i*		0.7043(3)	0.5357(3)	–0.0889(2)	0.027(2)	0.037(2)	0.047(2)	0.005(1)	–0.015(1)	–0.025(2)
O(1)	2*i*		0.2031(2)	0.8357(2)	0.0229(2)	0.030(1)	0.054(2)	0.066(2)	0.006(1)	–0.017(1)	–0.044(2)
O(2)	2*i*		0.3169(2)	0.9333(2)	0.1096(2)	0.026(1)	0.056(2)	0.058(2)	0.008(1)	–0.016(1)	–0.040(2)
O(3)	2*i*		0.0092(3)	0.8561(3)	0.0631(3)	0.031(2)	0.098(3)	0.107(3)	0.008(2)	–0.025(2)	–0.079(2)
O(4)	2*i*		0.5116(2)	0.8234(2)	0.0511(2)	0.032(1)	0.047(2)	0.060(2)	0.009(1)	–0.019(1)	–0.038(2)
O(5)	2*i*		0.6844(3)	0.7485(3)	0.0615(3)	0.040(2)	0.088(2)	0.098(3)	0.021(2)	–0.038(2)	–0.073(2)
O(6)	2*i*		0.4119(2)	0.7448(2)	–0.0581(2)	0.030(1)	0.044(2)	0.056(2)	0.011(1)	–0.021(1)	–0.033(1)
O(7)	2*i*		0.4263(2)	1.0054(2)	–0.1143(2)	0.045(2)	0.041(2)	0.055(2)	0.002(1)	–0.020(1)	–0.024(1)
O(8)	2*i*		0.7624(3)	0.8569(3)	0.1600(3)	0.047(2)	0.082(2)	0.081(3)	0.013(2)	–0.030(2)	–0.055(2)
O(9)	2*i*		0.9325(3)	0.7259(3)	0.9768(3)	0.050(2)	0.071(2)	0.093(3)	0.005(2)	–0.020(2)	–0.057(2)
O(10)	2*i*	0.5	0.9430(5)	0.4803(3)	0.5662(6)	0.099(7)	0.111(8)	0.126(9)	0.051(6)	–0.022(6)	–0.020(7)

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