Crystal structure of 1-cyclopropyl-6-fluoro-8-methoxy-7-(3-methyl-1-piperazinyl)-1,4-dihydro-4-oxo-3-quinolinecarboxylic acid hydrochloride, $[C_{19}H_{23}FN_3O_4]Cl$

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Discussion

In the title crystal structure, the bicyclic quinoline ring system is essentially planar, whereas the piperazine ring has a chair conformation (figure, top). The oxygen atom O2 of carboxylate group of quinolones is protonated. The terminal nitrogen atom N3 of the piperazine group is also protonated. The positive charge of H₂-Gat is neutralized by one chloride anion. The cyclopropyl group plane (C4, C5 and C6) is not orthogonal to the mean plane of the quinolone system; the corresponding dihedral angle is 61.9°. The –OCH₃ group is sticking out of the plane with torsion angles of 100.3(3)° for C13–O4–C12–C7. The oxygen atom O2 of carboxylate group of quinolones is protonated, and this proton is bonded to the oxygen O1 of the carbonyl group via intramolecular hydrogen O2–DH2···O1, forming a six membered pseudo-ring (O1/C9/C2/C1/O2/H2). The intermolecular hydrogen bond N3–H3···Cl1ⁱ (symmetry code i: $-x+1/2, y+1/2, -z+1/2$) and N3–H3B···Cl1ⁱⁱ (symmetry code ii: $x-1, y+1, z$) assembly the residues to form a 3D network (figure, bottom).

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.14 × 0.18 × 0.50 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	2.31 cm ⁻¹
Diffraction, scan mode:	Bruker SMART CCD, ϕ/ω
$2\theta_{\max}$:	52.02°
$N(hkl)_{\text{measured}}, N(hkl)_{\text{unique}}$:	11883, 3887
Criterion for $I_{\text{obs}}, N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2416
$N(\text{param})_{\text{refined}}$:	257
Programs:	SHELXS-97 [1], SHELXL-97 [2], SHELXTL [3]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(3A)	4e	-0.1962	0.9796	0.2466	0.078
H(3B)	4e	-0.1231	1.1318	0.2325	0.078
H(2)	4e	0.7403	0.8490	0.0286	0.121
H(3)	4e	0.4279	0.6398	-0.0382	0.063
H(4)	4e	0.1823	0.5374	0.0086	0.067
H(5A)	4e	0.2569	0.7473	-0.0854	0.094
H(5B)	4e	0.1579	0.5867	-0.0888	0.094
H(6A)	4e	0.1285	0.8945	-0.0203	0.083
H(6B)	4e	0.0295	0.7340	-0.0237	0.083
H(10)	4e	0.5013	1.0418	0.1541	0.073
H(13A)	4e	0.1784	0.4839	0.1213	0.126
H(13B)	4e	0.0342	0.5001	0.1225	0.126
H(13C)	4e	0.1161	0.5934	0.1681	0.126
H(15A)	4e	0.0021	0.9687	0.1097	0.085
H(15B)	4e	0.0016	1.1382	0.1463	0.085

Abstract

$C_{19}H_{23}ClFN_3O_4$, monoclinic, $P12_1/n1$ (no. 14), $a = 10.833(4)$ Å, $b = 7.683(2)$ Å, $c = 23.976(8)$ Å, $\beta = 91.11(2)^\circ$, $V = 1995.1$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.050$, $wR_{\text{ref}}(F^2) = 0.157$, $T = 296$ K.

Source of material

Gatifloxacin (HGat, 0.8 mmol, 0.3004 g) and FeCl₃ (0.5 mmol, 0.0650 g) were thoroughly mixed and placed into autoclave. After addition of 5 mL of EtOH and 15 mL of H₂O, the pH value of the solution was adjusted to 6.0. The autoclave was heated to 120 °C for 3 days to obtain pale yellow crystals of the title compound.

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Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(16A)	4e	-0.1901	1.0054	0.1519	0.091
H(16B)	4e	-0.1280	0.8301	0.1713	0.091
H(17)	4e	-0.0224	0.8169	0.2648	0.080
H(18A)	4e	-0.0267	1.1320	0.3258	0.134

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(18B)	4e	0.0362	0.9604	0.3473	0.134
H(18C)	4e	-0.1076	0.9689	0.3395	0.134
H(19A)	4e	0.1046	1.1283	0.2405	0.078
H(19B)	4e	0.1688	0.9542	0.2595	0.078

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Cl(1)	4e	0.88042(7)	0.4242(1)	0.22628(4)	0.0750(5)	0.0897(6)	0.0920(6)	-0.0022(4)	0.0201(4)	0.0065(5)
F(1)	4e	0.3214(2)	1.1250(3)	0.21166(7)	0.080(1)	0.101(1)	0.091(1)	-0.016(1)	0.0005(9)	-0.041(1)
N(1)	4e	0.3226(2)	0.7200(3)	0.02170(8)	0.043(1)	0.053(1)	0.051(1)	-0.0046(9)	-0.0094(9)	0.003(1)
N(2)	4e	0.1022(2)	0.9448(3)	0.18070(8)	0.052(1)	0.076(2)	0.048(1)	0.007(1)	-0.009(1)	0.000(1)
N(3)	4e	-0.1249(2)	1.0148(3)	0.23152(9)	0.055(1)	0.079(2)	0.061(1)	0.002(1)	0.002(1)	0.003(1)
O(1)	4e	0.6433(2)	0.9412(3)	0.08002(8)	0.047(1)	0.086(2)	0.089(1)	-0.015(1)	-0.012(1)	-0.002(1)
O(2)	4e	0.7528(2)	0.8061(3)	-0.0021(1)	0.049(1)	0.086(2)	0.108(2)	-0.007(1)	0.003(1)	0.005(1)
O(3)	4e	0.6461(2)	0.6565(3)	-0.06480(9)	0.065(1)	0.085(2)	0.074(1)	0.005(1)	0.007(1)	0.005(1)
O(4)	4e	0.1165(2)	0.7133(3)	0.09478(7)	0.048(1)	0.085(1)	0.061(1)	-0.0152(9)	-0.0035(8)	-0.002(1)
C(1)	4e	0.6489(2)	0.7378(4)	-0.0223(1)	0.049(2)	0.061(2)	0.074(2)	0.002(1)	-0.002(1)	0.019(2)
C(2)	4e	0.5393(2)	0.7701(3)	0.0131(1)	0.046(1)	0.051(2)	0.062(2)	-0.001(1)	-0.006(1)	0.013(1)
C(3)	4e	0.4290(2)	0.7024(3)	-0.0050(1)	0.051(2)	0.053(2)	0.054(1)	0.001(1)	-0.004(1)	0.006(1)
C(4)	4e	0.2114(2)	0.6496(4)	-0.0055(1)	0.047(1)	0.062(2)	0.057(2)	-0.009(1)	-0.010(1)	-0.002(1)
C(5)	4e	0.1947(3)	0.6794(5)	-0.0666(1)	0.058(2)	0.118(3)	0.059(2)	-0.014(2)	-0.014(1)	-0.010(2)
C(6)	4e	0.1149(2)	0.7709(4)	-0.0260(1)	0.052(2)	0.085(2)	0.069(2)	-0.004(1)	-0.021(1)	0.006(2)
C(7)	4e	0.3216(2)	0.8083(3)	0.07298(9)	0.045(1)	0.046(1)	0.050(1)	-0.001(1)	-0.010(1)	0.006(1)
C(8)	4e	0.4310(2)	0.8911(3)	0.0917(1)	0.047(1)	0.050(2)	0.056(2)	-0.003(1)	-0.013(1)	0.007(1)
C(9)	4e	0.5446(2)	0.8707(3)	0.0624(1)	0.044(1)	0.054(2)	0.063(2)	-0.004(1)	-0.010(1)	0.012(1)
C(10)	4e	0.4290(2)	0.9907(4)	0.1404(1)	0.050(2)	0.065(2)	0.067(2)	-0.009(1)	-0.011(1)	-0.003(1)
C(11)	4e	0.3229(3)	1.0128(4)	0.1675(1)	0.063(2)	0.061(2)	0.059(2)	-0.007(1)	-0.011(1)	-0.009(1)
C(12)	4e	0.2173(2)	0.8159(3)	0.1065(1)	0.042(1)	0.056(2)	0.052(1)	-0.004(1)	-0.009(1)	0.002(1)
C(13)	4e	0.1108(3)	0.5598(4)	0.1296(1)	0.076(2)	0.088(2)	0.088(2)	-0.032(2)	0.013(2)	0.003(2)
C(14)	4e	0.2129(2)	0.9275(3)	0.1528(1)	0.050(1)	0.062(2)	0.051(1)	0.004(1)	-0.006(1)	0.005(1)
C(15)	4e	-0.0022(2)	1.0121(5)	0.1476(1)	0.060(2)	0.099(2)	0.053(2)	0.015(2)	-0.005(1)	0.009(2)
C(16)	4e	-0.1211(3)	0.9559(5)	0.1728(1)	0.056(2)	0.115(3)	0.056(2)	0.004(2)	-0.010(1)	0.000(2)
C(17)	4e	-0.0188(3)	0.9444(4)	0.2653(1)	0.067(2)	0.082(2)	0.050(2)	0.000(2)	-0.006(1)	0.007(1)
C(18)	4e	-0.0303(3)	1.0072(5)	0.3249(1)	0.092(2)	0.117(3)	0.058(2)	0.001(2)	0.001(2)	-0.001(2)
C(19)	4e	0.0988(2)	1.0024(4)	0.2390(1)	0.063(2)	0.080(2)	0.051(2)	0.005(2)	-0.010(1)	0.000(1)

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