Crystal structure of bis[4-(5-amino-3-carboxy-1-cyclopropyl-6,8-difluoro-4-oxo-1,4-dihydroquinoline-7-yl)-2,6-dimethylpiperazin-1-ium]benzene-1,4-dicarboxylate, $(C_{19}H_{23}F_2N_4O_3)_2^+(C_8H_4O_4)^-$, $C_{23}H_{25}F_2N_4O_5$

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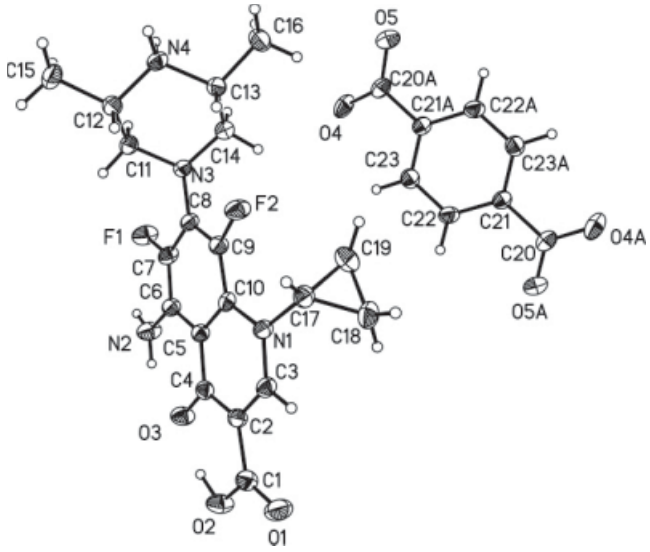

Abstract

$C_{23}H_{25}F_2N_4O_5$, triclinic, $P\bar{1}$ (no. 2), a = 7.397(1) Å, b = 9.649(1) Å, c = 15.361(2) Å, α = 89.30(1)°, β = 79.57(1)°, γ = 86.40(1)°, V = 1076.1 Å^3, Z = 2, $R_{gt}(F)$ = 0.0503, $wR_{ref}(F^2)$ = 0.1532, T = 296 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.20×0.23×0.24 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.16 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	15445, 3785
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2224
$N(param)_{refined}$:	342
Programs:	SHELX [6]

Source of material

A mixture of $Mg(NO_3)_2{\cdot}2H_2O$ (0.2 mmol), sparfloxacin (0.4 mmol), 1,4-benzenedicarboxylic acid (0.20 mmol), sodium hydroxide (0.5 mmol) and water (15 ml) was stirred for 15 min. The mixture was transferred to a 25 ml Teflon reactor and kept at 443 K for 96h under autogenous pressure. After cooling, colourless single crystals of title compound suitable for X-ray analysis were obtained.

Discussion

Sparfloxacin (H-Spar), $C_{19}H_{22}F_2N_4O_3$ (systematic name 5-amino-1-cyclopropyl-7-(3,5-dimethylpiperazin-1-yl)-6,8-difluoro-4-oxo-quinoline -3-carboxylic acid) is a member of the class of quinolones derivative [1]. The crystal structures of its anionic complexes with nickel, copper, cadmium and zinc have been reported [2-5]. Trying to prepare a corresponding magnesium(II) complex the title compound was obtained. The title structure is built up from mono protonated sparfloxacin cations and 1,4-benzenedicarboxylate anions in the ratio 2:1. There are intermolecular N–H···O hydrogen bonds from the positively charged piperazinium ring towards the carboxylate groups of the counter anion. Additionally there are intramolecular O–H···O and N–H···O bonds towards the carbonyl oxygen atom.

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

Atom	Site	x	y	z	U_{iso}
H(8)	2*i*	0.7986	0.2593	0.9362	0.046
H(13A)	2*i*	1.1635	0.9851	0.8254	0.049
H(13B)	2*i*	1.1220	1.0752	0.7447	0.049
H(14A)	2*i*	0.9472	0.9523	0.6365	0.051
H(14B)	2*i*	0.9097	0.7967	0.6583	0.051
H(15A)	2*i*	0.6732	0.2496	0.8024	0.063
H(15B)	2*i*	0.8418	0.1819	0.7285	0.063
H(17A)	2*i*	1.5979	0.9991	0.6757	0.085
H(17B)	2*i*	1.5178	1.0182	0.7768	0.085
H(17C)	2*i*	1.4503	1.1202	0.7074	0.085
H(18A)	2*i*	1.1263	0.9095	0.4854	0.076
H(18B)	2*i*	1.0710	0.7552	0.4964	0.076
H(18C)	2*i*	1.2789	0.7876	0.4700	0.076
H(19A)	2*i*	0.8734	0.4026	0.6562	0.068
H(19B)	2*i*	0.7048	0.4702	0.7302	0.068
H(20)	2*i*	0.6021	0.5677	0.3556	0.051
H(23)	2*i*	0.4401	0.3706	0.3844	0.051
H(1)	2*i*	1.046(4)	0.343(3)	0.771(2)	0.052(9)
H(2)	2*i*	1.430(4)	0.894(3)	0.581(2)	0.050(9)
H(3)	2*i*	1.290(4)	1.013(4)	0.594(2)	0.07(1)
H(4)	2*i*	1.219(3)	0.734(3)	0.623(2)	0.026(7)
H(5)	2*i*	1.369(3)	0.832(3)	0.728(2)	0.042(8)
H(6)	2*i*	0.587(6)	0.466(4)	1.168(3)	0.11(2)
H(7)	2*i*	0.695(5)	0.949(1)	1.050(2)	0.08(1)
H(9)	2*i*	0.652(4)	0.808(3)	1.099(1)	0.06(1)

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

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	2*i*	0.9356(4)	0.8053(3)	0.8206(2)	0.037(2)	0.032(2)	0.037(2)	–0.007(1)	–0.002(1)	0.000(1)
C(2)	2*i*	0.8613(3)	0.5779(3)	0.8828(2)	0.032(2)	0.030(2)	0.038(2)	–0.005(1)	–0.005(1)	–0.004(1)
C(3)	2*i*	0.7816(3)	0.6397(3)	0.9660(2)	0.034(2)	0.034(2)	0.033(2)	–0.002(1)	–0.003(1)	–0.002(1)
C(4)	2*i*	0.9414(4)	0.6616(3)	0.8145(2)	0.036(2)	0.037(2)	0.031(2)	–0.003(1)	0.003(1)	–0.008(1)
C(5)	2*i*	0.7780(4)	0.7868(3)	0.9745(2)	0.043(2)	0.040(2)	0.033(2)	–0.006(1)	0.001(1)	–0.006(1)
C(6)	2*i*	0.7212(4)	0.4077(3)	1.0258(2)	0.037(2)	0.040(2)	0.035(2)	–0.003(1)	–0.007(1)	0.003(1)
C(7)	2*i*	0.7109(4)	0.5532(3)	1.0399(2)	0.036(2)	0.044(2)	0.033(2)	–0.005(1)	–0.005(1)	–0.002(1)
C(8)	2*i*	0.7936(4)	0.3550(3)	0.9437(2)	0.042(2)	0.030(2)	0.045(2)	–0.004(1)	–0.012(1)	0.003(1)
C(9)	2*i*	0.9130(4)	0.3638(3)	0.7875(2)	0.044(2)	0.037(2)	0.037(2)	–0.002(1)	–0.002(1)	–0.008(1)
C(10)	2*i*	1.1773(4)	0.8224(3)	0.6011(2)	0.041(2)	0.036(2)	0.034(2)	–0.006(1)	–0.006(1)	0.000(1)
C(11)	2*i*	0.8532(4)	0.8610(3)	0.9012(2)	0.050(2)	0.027(2)	0.045(2)	–0.006(1)	0.002(2)	–0.005(1)
C(12)	2*i*	1.3381(4)	0.9316(3)	0.7099(2)	0.044(2)	0.037(2)	0.037(2)	–0.006(1)	–0.008(1)	0.001(1)
C(13)	2*i*	1.1544(4)	0.9819(3)	0.7633(2)	0.049(2)	0.034(2)	0.041(2)	–0.012(1)	–0.008(1)	–0.000(1)
C(14)	2*i*	0.9965(4)	0.8673(3)	0.6602(2)	0.043(2)	0.046(2)	0.040(2)	–0.008(1)	–0.006(1)	0.002(1)
C(15)	2*i*	0.7869(4)	0.2637(3)	0.7616(2)	0.054(2)	0.047(2)	0.058(2)	–0.010(2)	–0.008(2)	–0.015(2)
C(16)	2*i*	0.6513(4)	0.3069(4)	1.0958(2)	0.048(2)	0.046(2)	0.046(2)	–0.003(2)	–0.011(2)	0.006(2)
C(17)	2*i*	1.4899(4)	1.0259(3)	0.7182(2)	0.047(2)	0.064(2)	0.063(2)	–0.022(2)	–0.010(2)	–0.005(2)
C(18)	2*i*	1.1620(4)	0.8183(3)	0.5044(2)	0.049(2)	0.064(2)	0.039(2)	–0.001(2)	–0.011(2)	–0.006(2)
C(19)	2*i*	0.8068(5)	0.4010(3)	0.7167(2)	0.065(2)	0.061(2)	0.042(2)	0.005(2)	–0.012(2)	–0.009(2)
C(20)	2*i*	0.5612(4)	0.5412(3)	0.4139(2)	0.049(2)	0.037(2)	0.039(2)	–0.004(1)	–0.004(1)	0.009(1)
C(21)	2*i*	0.5978(4)	0.6205(3)	0.4823(2)	0.036(2)	0.030(2)	0.042(2)	–0.000(1)	–0.006(1)	0.003(1)
C(22)	2*i*	0.6917(4)	0.7544(3)	0.4634(2)	0.040(2)	0.034(2)	0.057(2)	–0.005(1)	–0.011(2)	0.007(2)
C(23)	2*i*	0.4641(4)	0.4223(3)	0.4312(2)	0.052(2)	0.036(2)	0.042(2)	–0.001(1)	–0.012(2)	0.000(1)
F(1)	2*i*	1.0438(2)	0.6044(2)	0.7395(1)	0.064(1)	0.042(1)	0.041(1)	–0.0098(9)	0.0143(9)	–0.0070(8)
F(2)	2*i*	0.8400(3)	1.0022(2)	0.9090(1)	0.090(1)	0.031(1)	0.058(1)	–0.0070(9)	0.013(1)	–0.0079(8)
N(1)	2*i*	1.3177(4)	0.9216(3)	0.6147(2)	0.039(2)	0.035(2)	0.042(2)	–0.009(1)	–0.003(1)	0.005(1)
N(2)	2*i*	1.0104(3)	0.8907(2)	0.7518(2)	0.048(1)	0.041(2)	0.034(1)	–0.016(1)	–0.004(1)	0.002(1)
N(3)	2*i*	0.8579(3)	0.4331(2)	0.8735(1)	0.036(1)	0.032(1)	0.034(1)	–0.004(1)	–0.002(1)	–0.001(1)
N(4)	2*i*	0.7028(4)	0.8565(3)	1.0508(2)	0.087(2)	0.041(2)	0.041(2)	–0.010(2)	0.014(2)	–0.010(1)
O(1)	2*i*	0.6556(3)	0.1830(2)	1.0846(2)	0.083(2)	0.045(2)	0.060(2)	–0.010(1)	–0.008(1)	0.012(1)
O(2)	2*i*	0.5797(3)	0.3635(3)	1.1739(1)	0.075(2)	0.057(2)	0.042(1)	–0.002(1)	0.004(1)	0.010(1)
O(3)	2*i*	0.6402(3)	0.6038(2)	1.1162(1)	0.070(2)	0.050(1)	0.035(1)	–0.004(1)	0.005(1)	–0.001(1)
O(4)	2*i*	0.6599(3)	0.8458(2)	0.5232(2)	0.048(1)	0.035(1)	0.086(2)	–0.009(1)	0.000(1)	–0.012(1)
O(5)	2*i*	0.7903(3)	0.7705(2)	0.3898(2)	0.076(2)	0.061(2)	0.055(2)	–0.027(1)	–0.002(1)	0.015(1)

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