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Sparfloxacin, an antibacterial drug[1]

A. Sivalakshmidevi, K. Vyas* and G. Om Reddy

Dr. Reddy's Research Foundation, 7-1-27 Ameerpet, Hyderabad 500 016, India
Correspondence e-mail: vyas11@usa.net

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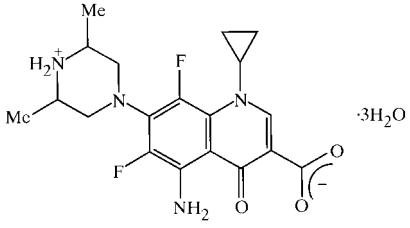
The title compound, sparfloxacin or *cis*-5-amino-1-cyclopropyl-7-(3,5-dimethylpiperazin-1-yl)-6,8-difluoro-1,4-dihydro-4-oxoquinoline-3-carboxylic acid trihydrate, $C_{19}H_{22}F_2N_4O_3\cdot3H_2O$, is an antibacterial drug. The molecule, which crystallizes as a trihydrate, is in the zwitterionic form in the solid state. Hydrogen bonds stabilize the molecules in the lattice.

Comment

Fluorinated quinolones are extensively used as antibacterial agents. X-ray diffraction studies of the title compound, (I), have been undertaken as part of a structural elucidation project. All the bond lengths and angles are normal (Allen *et al.*, 1987). The piperazine ring assumes a chair form as evidenced by the torsion angles. The relevant asymmetry parameters are $\Delta C2$ = 1.8, 1.51 and 1.6, and ΔC_s = 1.06, 2.21 and 3.08 (Duax *et al.*, 1976). The weight loss of 11.5% in the thermogravimetric analysis experiment indicates the presence of three molecules of water. The N2 atom of the amino group is involved in intramolecular hydrogen bonding with the O3 atom of the carbonyl group of the quinolone moiety.



(I)

Three intermolecular hydrogen-bonding interactions *viz.*, molecule–molecule, molecule–water and water–water, stabilize the molecules in the lattice. Donating its two H atoms, the protonated amino piperazine N4 atom is involved in hydrogen bonding with the quinolone carbonyl O3 atom and the the O2 atom of the caboxylate group. The amino N2 atom interacts with the O1 atom of the carboxylate group. Both the water molecules O4 and O6 have hydrogen-bond interactions with the carboxylate O1 and O2′ atoms. The water O5 atom donates a H atom to the water O4 and O6 atoms and, in addition, accepts a H atom from the piperazine N4 atom to form a hydrogen bond. Hydrogen-bonding parameters are presented in Table 2.

Experimental

Crystals of sparfloxacin suitable for X-ray diffraction were grown from a mixture of ethyl acetate, chloroform and acetone.

Crystal data

$C_{19}H_{22}F_2N_4O_3\cdot3H_2O$	D_x = 1.375 Mg m^{-3}
M_r = 446.45	Cu $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 25 reflections
a = 11.850 (3) Å	θ = 25.0–30.9°
b = 10.913 (4) Å	μ = 0.974 mm^{-1}
c = 17.506 (2) Å	T = 298.2 K
β = 107.76 (1)°	Needle, colourless
V = 2156.0 (8) Å^3	0.50 × 0.50 × 0.25 mm
Z = 4	

Data collection

Rigaku AFC-7*S* diffractometer	R_{int} = 0.036
ω–2θ scans	θ_{max} = 70.12°
Absorption correction: ψ scan (North *et al.*, 1968)	$h = 0 \rightarrow 14$
T_{min} = 0.684, T_{max} = 0.784	$k = 0 \rightarrow 13$
4480 measured reflections	$l = -21 \rightarrow 20$
4479 independent reflections	3 standard reflections every 150 reflections
4260 reflections with $I > 1.2\sigma(I)$	intensity decay: −0.30%

Refinement

Refinement on F	$w = 1/[\sigma^2(F_o) + 0.00016\|F_o\|^2]$
R = 0.059	$(\Delta/\sigma)_{max}$ = 0.004
wR = 0.108	$\Delta\rho_{max}$ = 0.30 e Å^{-3}
S = 1.987	$\Delta\rho_{min}$ = −0.27 e Å^{-3}
3550 reflections	Extinction correction: Zachariasen (1967)
393 parameters	Extinction coefficient: 6 (1) × 10^{-6}
All H-atom parameters refined	

Table 1
Selected geometric parameters (Å, °).

F1—C6	1.356 (3)	C2—C19	1.518 (3)
F2—C8	1.360 (3)	C3—C4	1.462 (3)
O1—C19	1.250 (3)	C4—C5	1.430 (3)
O2—C19	1.257 (3)	C4—C9	1.429 (3)
O3—C3	1.245 (3)	C5—C6	1.393 (3)
N1—C1	1.349 (3)	C6—C7	1.388 (3)
N1—C5	1.398 (3)	C7—C8	1.385 (3)
N1—C16	1.453 (3)	C8—C9	1.389 (3)
N2—C9	1.348 (3)	C10—C11	1.510 (3)
N3—C7	1.391 (3)	C11—C15	1.509 (3)
N3—C10	1.456 (3)	C12—C13	1.512 (4)
N3—C13	1.452 (3)	C12—C14	1.515 (4)
N4—C11	1.504 (3)	C16—C17	1.481 (4)
N4—C12	1.496 (3)	C16—C18	1.497 (4)
C1—C2	1.354 (3)	C17—C18	1.505 (5)
C2—C3	1.440 (3)		

[1] Publication No. 125 from the DRF.

C1—N1—C5	119.3 (2)	N3—C7—C8	123.0 (2)
C1—N1—C16	118.6 (2)	C6—C7—C8	116.9 (2)
C5—N1—C16	122.1 (2)	F2—C8—C7	118.9 (2)
C7—N3—C10	120.9 (2)	F2—C8—C9	116.4 (2)
C7—N3—C13	121.2 (2)	C7—C8—C9	124.7 (2)
C10—N3—C13	114.1 (2)	N2—C9—C4	122.8 (2)
C11—N4—C12	113.3 (2)	N2—C9—C8	120.0 (2)
N1—C1—C2	125.1 (2)	C4—C9—C8	117.1 (2)
C1—C2—C3	119.3 (2)	N3—C10—C11	110.5 (2)
C1—C2—C19	117.8 (2)	N4—C11—C10	108.1 (2)
C3—C2—C19	122.8 (2)	N4—C11—C15	109.6 (2)
O3—C3—C2	122.9 (2)	C10—C11—C15	112.7 (2)
O3—C3—C4	121.0 (2)	N4—C12—C13	107.8 (2)
C2—C3—C4	116.1 (2)	N4—C12—C14	110.0 (2)
C3—C4—C5	120.2 (2)	C13—C12—C14	112.2 (2)
C3—C4—C9	120.5 (2)	N3—C13—C12	109.3 (2)
C5—C4—C9	119.3 (2)	N1—C16—C17	119.4 (2)
N1—C5—C4	118.8 (2)	N1—C16—C18	118.7 (2)
N1—C5—C6	122.1 (2)	C17—C16—C18	60.7 (2)
C4—C5—C6	119.0 (2)	C16—C17—C18	60.1 (2)
F1—C6—C5	120.9 (2)	C16—C18—C17	59.1 (2)
F1—C6—C7	116.2 (2)	O1—C19—O2	125.5 (2)
C5—C6—C7	122.5 (2)	O1—C19—C2	116.5 (2)
N3—C7—C6	119.9 (2)	O2—C19—C2	118.0 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H23···O1	0.93 (5)	1.87 (5)	2.784 (3)	168 (4)
O4—H24···O2^{i}	1.00 (5)	1.92 (5)	2.845 (3)	152 (4)
O5—H27···O6^{ii}	0.89 (6)	1.94 (6)	2.810 (4)	168 (6)
O5—H28···O4^{iii}	0.80	2.04	2.781 (4)	155
O6—H25···O2^{i}	0.92 (6)	2.06 (6)	2.959 (4)	163 (5)
O6—H26···O1	0.95 (5)	1.91 (5)	2.818 (3)	159 (5)
N2—H2···O3	0.92 (3)	1.85 (3)	2.616 (2)	139 (3)
N2—H3···O1^{iv}	0.86 (3)	2.11 (3)	2.890 (3)	151 (3)
N4—H7···O5	0.94 (3)	1.91 (3)	2.829 (3)	166 (3)
N4—H8···O3^{v}	0.81 (4)	2.01 (4)	2.733 (2)	148 (4)
N4—H8···O2^{v}	0.81 (4)	2.38 (4)	2.985 (3)	132 (3)

Symmetry codes: (i) $\frac{5}{2}-x, y-\frac{1}{2}, \frac{3}{2}-z$; (ii) $x-\frac{3}{2}, \frac{1}{2}-y, z-\frac{1}{2}$; (iii) $1-x, -y, 1-z$; (iv) $x-\frac{1}{2}, \frac{1}{2}-y, z-\frac{1}{2}$; (v) $x-1, y, z$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1994); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *teXsan* (Molecular Structure Corporation, 1995); program(s) used to solve structure: *SIR*92 (Altomare *et al.* 1993); program(s) used to refine structure: *teXsan*; software used to prepare material for publication: *teXsan*.

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