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1-Cyclopropyl-6-fluoro-7-(4-formylpiperazin-1-yl)-4-oxo-1,4-dihydroquinoline-3-carboxylic acid

Xian-Wen Li,[a,b] Feng Zhi,[a] Jian-Hua Shen[a] and Yi-Qiao Hu[a]*

[a]School of Life Sciences, Nanjing University, Nanjing 210093, People's Republic of China, and [b]Department of Biochemistry, Fujian Institute of Education, Fuzhou 350001, Fujian, People's Republic of China

Correspondence e-mail: hu_yiqiao@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.054
wR factor = 0.156
Data-to-parameter ratio = 13.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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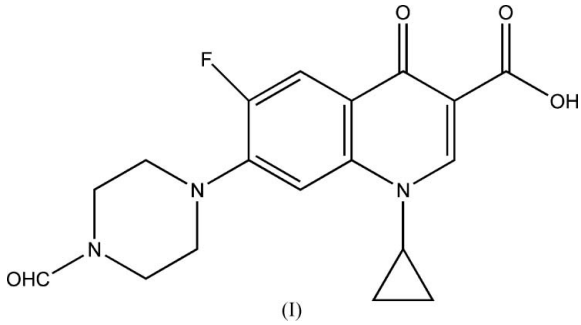
In the title compound, $C_{18}H_{17}FN_3O_4$, the piperazinyl ring adopts a chair conformation. The cyclopropyl rings is not coplanar with the quinolone ring system.

Comment

In recent years, a number of highly potent broad-spectrum antibacterial agents have been synthesized, and several have been introduced into clinical use. Ciprofloxacin [1-cyclopropyl-6-fluoro-4-oxo-7-(1-piperazinyl)-1,4-dihydroquinoline-3-carboxylic acid] is one of the most active quinolone antibacterial agents. The crystal structure of the title derivative compound of ciprofloxacin, (I), is reported here.



(I)

All bond lengths and angles in compound (I) (Fig. 1) are in normal ranges (Allen *et al.*, 1987), and are comparable with the corresponding values observed in a similar compound, ciprofloxacin (Turel *et al.*, 1997).

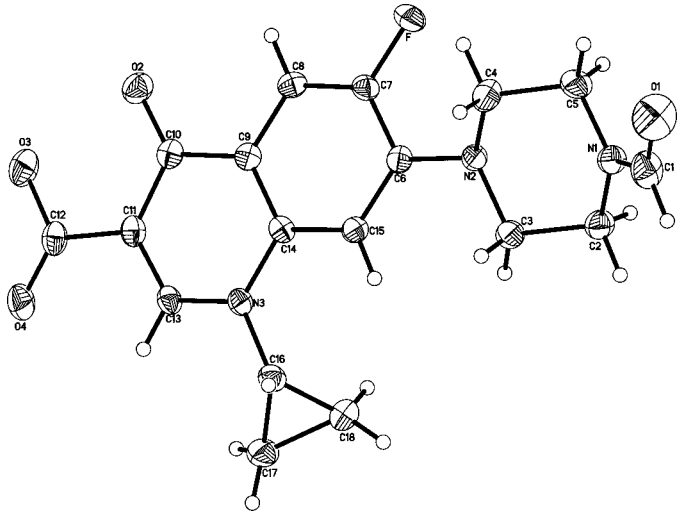


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

In the title structure (Fig. 1), the piperazinyl group adopts a chair conformation. The cyclopropyl ring is not coplanar with the quinolone ring system. The geometry of the rigid quinolone ring system is similar to that found in a reported structure (Turel *et al.*, 1997).

Experimental

The title compound was prepared as follows. Ciprofloxacin (80 mg) was dissolved in formamide (40 ml) and the solution was heated under reflux for 30 min at 353 K. The reaction mixture was cooled and allowed to stand, resulting in the formation of yellow needle-like crystals of (I) (m.p. 565.9–567.5 K). Analysis, found: C 60.1, H 5.1, N 11.6%; calculated for $C_{18}H_{18}FN_3O_4$: C 60.2, H 5.0, N 11.7%.

Crystal data

$C_{18}H_{17}FN_3O_4$
M_r = 358.35
Triclinic, $P\overline{1}$
a = 8.4140 (17) Å
b = 9.5130 (19) Å
c = 10.497 (2) Å
α = 102.57 (3)°
β = 96.58 (3)°
γ = 97.08 (3)°
V = 805.1 (3) Å^3
Z = 2
D_x = 1.478 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 9–13°
μ = 0.11 mm^{-1}
T = 293 (2) K
Needle, yellow
0.5 × 0.2 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (*XCAD4*; Harms & Wocadlo, 1995)
T_{min} = 0.970, T_{max} = 0.973
3382 measured reflections
3157 independent reflections
1824 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 26.0°
$h = 0 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -12 \rightarrow 12$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.156
S = 1.05
3157 reflections
235 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.21P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

H atoms were positioned geometrically and treated as riding atoms, with C—H distances in the range 0.93–0.98 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXTL*.

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1-Cyclopropyl-6-fluoro-7-(4-formylpiperazin-1-yl)-4-oxo-1,4-dihydroquinoline-3-carboxylic acid. Corrigendum

Xian-Wen Li,[a,b] Feng Zhi,[b] Jian-Hua Shen[b] and Yi-Qiao Hu[b]*

[a]School of Life Sciences, Nanjing University, Nanjing 210093, People's Republic of China, and [b]Department of Biochemistry, Fujian Institute of Education, Fuzhou 350001, Fujian, People's Republic of China

Correspondence e-mail: hu_yiqiao@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.050
wR factor = 0.140
Data-to-parameter ratio = 13.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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The crystal structure of the title compound, $C_{18}H_{18}FN_3O_4$, was published [Li *et al.* (2005). *Acta Cryst.* E**61**, o2235–o2236] with an error in the chemical formula and without location of the carboxyl H atom. This has now been corrected. The missing H atom was located and refined. This H atom is involved in an intramolecular O—H···O hydrogen bond with the carbonyl O atom.

Experimental

Crystal data

$C_{18}H_{18}FN_3O_4$
M_r = 359.35
Triclinic, $P\overline{1}$
a = 8.414 (2) Å
b = 9.513 (2) Å
c = 10.497 (2) Å
α = 102.57 (3)°
β = 96.58 (3)°
γ = 97.08 (3)°
V = 805.1 (3) Å^3
Z = 2
D_x = 1.482 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 9–13°
μ = 0.11 mm^{-1}
T = 293 (2) K
Block, yellow
0.48 × 0.21 × 0.19 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (*XCAD4*; Harms & Wocadlo, 1995)
T_{min} = 0.947, T_{max} = 0.979
3379 measured reflections
3154 independent reflections
1822 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 26.0°
$h = 0 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -12 \rightarrow 12$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.140
S = 1.01
3154 reflections
238 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.035P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O2	0.90 (1)	1.69 (2)	2.514 (3)	151 (3)

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Harms, K. & Wocadlo, S. (1995) *XCAD4*. University of Marburg, Germany.
Li, X.-W., Zhi, F., Shen, J.-H. & Hu, Y.-Q. (2005). *Acta Cryst.* E**61**, o2235–o2236.