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7-Chloro-1-ethyl-6-fluoro-1,4-dihydro-4-oxo-quinoline-3-carboxylic acid

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Key indicators

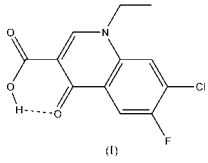
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.036
wR factor = 0.104
Data-to-parameter ratio = 13.8

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

The title compound, $C_{12}H_9ClFNO_3$, was synthesized from ethyl 2,4-dichloro-5-fluorobenzoylacetate. The quinoline ring has a same plane with the carboxyl group.

Comment

Norfloxacin [1-ethyl-6-fluoro-1,4-dihydro-4-oxo-7-(1-piperazinyl)-3-quinolinecarboxylic acid] is a fluorinated quinolone antibacterial agent (Koga *et al.*, 1980). Fluorinated quinolone compounds are characterized by having an F atom at the 6-position and a substituted amine group at the 7-position. The title compound, (I), is an intermediate in the synthesis of norfloxacin; the molecular structure of (I) is illustrated in Fig. 1. The quinoline ring has a same plane with the carboxyl group.In the crystal structure of (I),molecules are connected into two-dimensional layers,where are existing C—H···O and C—H···Cl hydrogen bonds.



(I)

Experimental

The title compound was prepared according to the method of Irikura (1978) from ethyl 2,4-dichloro-5-fluorobenzoylacetate. Condensation of ethyl 2,4-dichloro-5-fluorobenzoylacetate with triethyl orthoformate by refluxing in acetic anhydride produced ethyl 2-(2,4-dichloro-5-fluorobenzoyl)-3-ethoxyacrylate. The intermediate was reacted without further purification with ethanamine in methylene chloride to afford ethyl 2-(2,4-dichloro-5-fluorobenzoyl)-3-ethanaminoacrylate. This was cyclized by heating with sodium hydride to give 7-chloro-1-ethyl-6-fluoro-1,4-dihydro-3-ethoxycarbonyl-4-oxoquinoline. The ester was hydrolyzed by heating with aqueous KOH in tetrahydrofuran to give 7-chloro-1-ethyl-6-fluoro-1,4-dihydro-4-oxoquinoline-3-carboxylic acid. The crude product was purified by

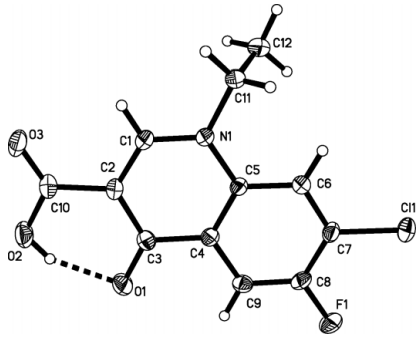


Figure 1
The molecular structure of (I), drawn with 30% probability ellipsoids. The intramolecular hydrogen bond is indicated by a dashed line.

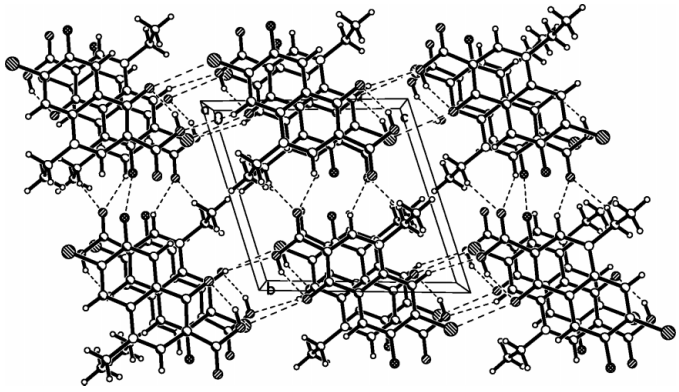


Figure 2
The crystal structure of (I), viewed along the *a* axis.

recrystallization from *N*,*N*-dimethylformamide. The product (20 mg) was dissolved in dichloromethane (20 ml) and the solution kept at room temperature for 6 d. Slow evaporation produced colorless single crystals of (I) suitable for X-ray analysis.

Crystal data

$C_{12}H_9ClFNO_3$
$M_r = 269.65$
Triclinic, $P\overline{1}$
$a = 7.145$ (3) Å
$b = 8.915$ (4) Å
$c = 9.375$ (4) Å
$\alpha = 71.896$ (6)°
$\beta = 80.025$ (7)°
$\gamma = 85.109$ (7)°
$V = 558.7$ (4) Å^3
$Z = 2$
$D_x = 1.603$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 792 reflections
$\theta = 3.7$–26.4°
$\mu = 0.35$ mm^{-1}
$T = 293$ (2) K
Block, colorless
0.36 × 0.34 × 0.28 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
$T_{min} = 0.809$, $T_{max} = 0.906$
3221 measured reflections
2273 independent reflections
1745 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 26.5°$
$h = -6 \rightarrow 8$
$k = -11 \rightarrow 10$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.104$
$S = 1.06$
2273 reflections
165 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0584P)^2 + 0.0322P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···Cl1^{i}	0.82	2.98	3.546 (2)	129
O2—H2···O1	0.82	1.79	2.550 (2)	155

Symmetry code: (i) $x, y, 1 + z$.

H atoms were positioned geometrically and refined with C—H = 0.93–0.98 Å and refined in a riding model with U_{iso} (H) = 1.2 U_{eq} (carrier).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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