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ISSN 2414-3146

Ethyl 1-methyl-2-oxo-1,2-dihydroquinoline-4-carboxylate

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Received 16 June 2017
Accepted 19 June 2017

Edited by J. Simpson, University of Otago, New Zealand

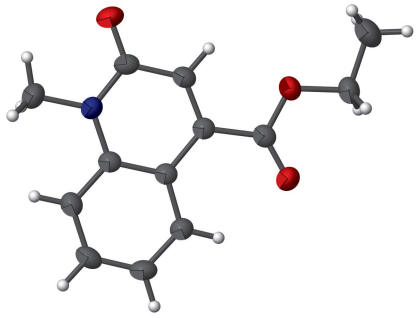
Keywords: crystal structure; quinoxaline; alkylation.

CCDC reference: 1556969

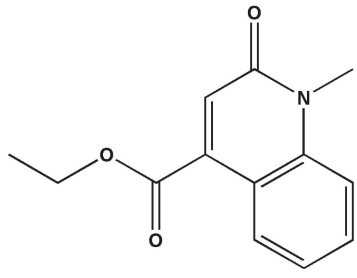
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{13}H_{13}NO_3$, lies on a mirror plane with an intramolecular C—H···O hydrogen bond enclosing an *S*(6) ring. In the crystal, weak C—H···O hydrogen bonds link the molecules into zigzag chains along the *a*-axis direction and π–π interactions, with a centroid-to-centroid distance of 3.7003 (2) Å, involving the pyridine and benzene rings of the oxoquinoline ring system, pack the molecules into parallel layers.

3D view



Chemical scheme



Structure description

Quinolone derivatives are a versatile class of nitrogen-containing heterocyclic compounds and they are useful intermediates in organic synthesis. They possess a broad spectrum of biological activities including anti-cancer (Elderfield & LeVon, 1960), anti-inflammatory (Ratheesh *et al.*, 2013) and antibacterial properties (Beena & Rawat, 2013; Chai *et al.*, 2011). Some quinoline derivatives have also been reported as corrosion inhibitors for steel in an acidic medium (Ebenso *et al.* 2010). Following on from our research in the field of substituted pyrido[2,3-*b*]pyrazine derivatives (Filali Baba *et al.*, 2016), we report here the synthesis of the title compound by the condensation reaction of iodomethane with ethyl 1,2-dihydro-2-oxoquinoline-4-carboxylate and its crystal structure.

The title compound lies on a mirror plane and crystallizes with one independent molecule in the asymmetric unit (Fig. 1). Only the hydrogen atoms of the methylene and methyl groups lie out of this plane. An intramolecular C5—H5···O2 hydrogen bond generates an *S*(6) ring motif. In the crystal, weak C8—H8···O1^{i} hydrogen bonds link the

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O2	0.93	2.24	2.892 (2)	126
C8—H8···O1^{i}	0.93	2.37	3.285 (2)	168

Symmetry code: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+\frac{1}{2}$.

molecules into zigzag chains along the *a*-axis direction (Table 1, Fig. 2). In addition, π–π interactions involving the pyridine and benzene rings of the oxoquinoline ring system stack the molecules into parallel layers [$Cg1\cdots Cg2$ = 3.7003 (2) Å, symmetry operations $1-x, -y, 1-z$; $1-x, 1-y, 1-z$; $1-x, -\frac{1}{2}+y, 1-z$; $1-x, \frac{1}{2}+y, 1-z$; $Cg1$ and $Cg2$ are the centroids of the N1/C1–C4/C9 and C4–C9 rings, respectively].

Synthesis and crystallization

A solution of ethyl 1,2-dihydro-2-oxoquinoline-4-carboxylate (1 g 4.6 mmol) in 15 ml of DMF was mixed with iodomethane (0.34 ml, 5.5 mmol), K_2CO_3 (0.82 g, 6 mmol) and TBAB(0.03 g, 0.1 mmol). The reaction mixture was stirred at room temperature in DMF for 6 h. After removal of salts by filtration, the DMF was evaporated under reduced pressure and the residue obtained was dissolved in dichloromethane. The organic phase was dried over Na_2SO_4 then concentrated *in vacuo*. The title compound was obtained after recrystallization from a dichloromethane/hexane (1/3) solvent mixture, yield = 81%.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{13}NO_3$
M_r	231.24
Crystal system, space group	Orthorhombic, *Pnma*
Temperature (K)	293
a, b, c (Å)	12.2269 (4), 6.7034 (3), 14.0817 (5)
V (Å^3)	1154.16 (8)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.78
Crystal size (mm)	0.16 × 0.12 × 0.04
Data collection	
Diffractometer	Rigaku Oxford Diffraction
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku Oxford Diffraction, 2015)
T_{min}, T_{max}	0.751, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6979, 1219, 1036
R_{int}	0.038
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.615
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.119, 1.05
No. of reflections	1219
No. of parameters	105
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.28, −0.15

Computer programs: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

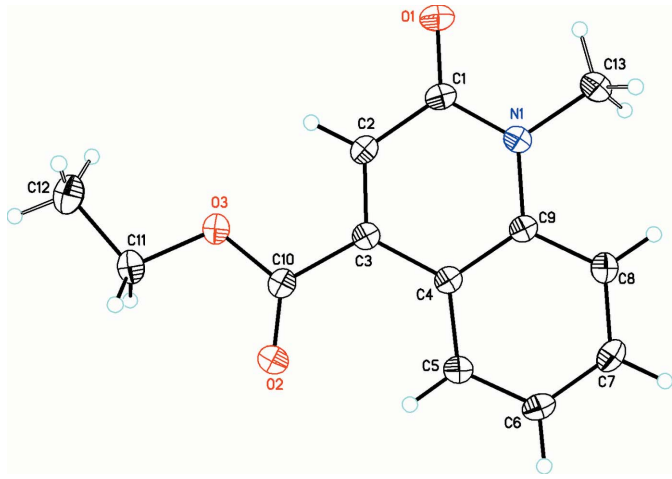


Figure 1
The structure of the title compound, showing the atom-numbering scheme, with displacement ellipsoids drawn at the 30% probability level.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms on the C12 and C13 methyl groups were generated using the PART −1 and AFIX 137 functions in *SHELXL*.

Acknowledgements

JPJ acknowledges the NSF–MRI program (grant No. CHE-1039027) for funds to purchase the X-ray diffractometer.

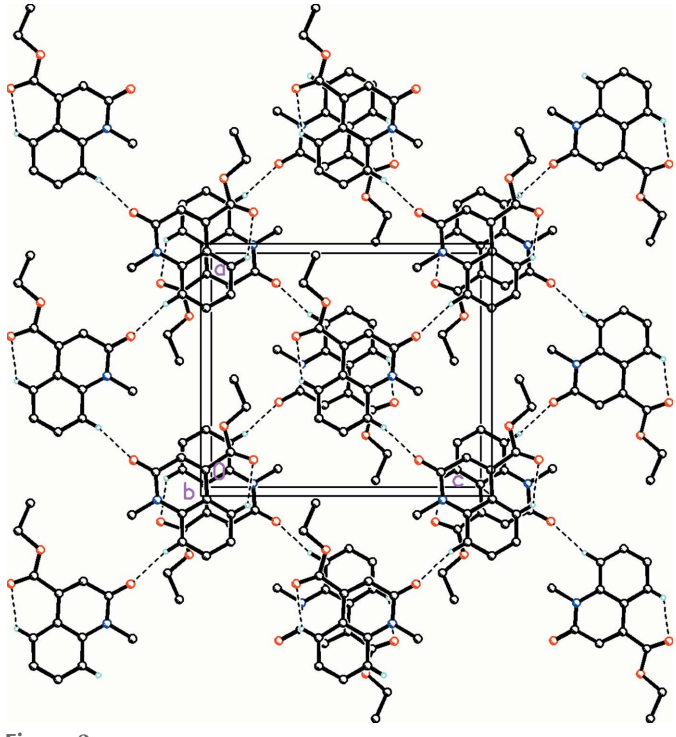


Figure 2
The packing of the title compound, viewed along the *b* axis. Dashed lines indicate both intra- and intermolecular hydrogen bonds. H atoms not involved in the packing have been omitted for clarity.

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full crystallographic data

IUCrData (2017). **2**, x170917 [https://doi.org/10.1107/S2414314617009178]

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Crystal data

$C_{13}H_{13}NO_3$
M_r = 231.24
Orthorhombic, *Pnma*
a = 12.2269 (4) Å
b = 6.7034 (3) Å
c = 14.0817 (5) Å
V = 1154.16 (8) Å^3
Z = 4
F(000) = 488
D_x = 1.331 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 2463 reflections
θ = 4.8–71.5°
μ = 0.78 mm^{-1}
T = 293 K
Plate, yellow
0.16 × 0.12 × 0.04 mm

Data collection

Rigaku Oxford Diffraction diffractometer
Radiation source: fine-focus sealed X-ray tube, Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 16.0416 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku Oxford Diffraction, 2015)
T_{min} = 0.751, T_{max} = 1.000
6979 measured reflections
1219 independent reflections
1036 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 71.5°, θ_{min} = 4.8°
h = −14→15
k = −5→8
l = −15→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.119
S = 1.05
1219 reflections
105 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.071P)^2 + 0.1102P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.36454 (11)	0.2500	0.25521 (9)	0.0580 (4)	
O2	0.36344 (10)	0.2500	0.67503 (9)	0.0544 (4)	
O3	0.22613 (9)	0.2500	0.57140 (8)	0.0446 (4)	
N1	0.52409 (11)	0.2500	0.33834 (9)	0.0352 (4)	
C1	0.41212 (13)	0.2500	0.33222 (12)	0.0386 (4)	
C2	0.35321 (13)	0.2500	0.42157 (12)	0.0367 (4)	
H2	0.2772	0.2500	0.4198	0.044*	
C3	0.40269 (13)	0.2500	0.50701 (11)	0.0313 (4)	
C4	0.52145 (12)	0.2500	0.51177 (11)	0.0303 (4)	
C5	0.58205 (13)	0.2500	0.59667 (12)	0.0370 (4)	
H5	0.5454	0.2500	0.6546	0.044*	
C6	0.69501 (14)	0.2500	0.59553 (13)	0.0439 (4)	
H6	0.7339	0.2500	0.6523	0.053*	
C7	0.75009 (14)	0.2500	0.50994 (13)	0.0447 (4)	
H7	0.8262	0.2500	0.5096	0.054*	
C8	0.69445 (13)	0.2500	0.42541 (12)	0.0390 (4)	
H8	0.7328	0.2500	0.3684	0.047*	
C9	0.57974 (13)	0.2500	0.42483 (11)	0.0312 (4)	
C10	0.33175 (13)	0.2500	0.59438 (12)	0.0346 (4)	
C11	0.14893 (14)	0.2500	0.64993 (14)	0.0491 (5)	
H11A	0.1592	0.3675	0.6891	0.059*	0.5
H11B	0.1592	0.1325	0.6891	0.059*	0.5
C12	0.03753 (17)	0.2500	0.60706 (19)	0.0834 (9)	
H12A	0.0232	0.3779	0.5790	0.125*	0.5
H12B	−0.0158	0.2236	0.6555	0.125*	0.5
H12C	0.0334	0.1485	0.5591	0.125*	0.5
C13	0.58549 (16)	0.2500	0.24866 (12)	0.0499 (5)	
H13A	0.6393	0.1458	0.2500	0.075*	0.5
H13B	0.6212	0.3764	0.2406	0.075*	0.5
H13C	0.5361	0.2278	0.1967	0.075*	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0450 (7)	0.0960 (12)	0.0330 (7)	0.000	−0.0106 (5)	0.000
O2	0.0409 (7)	0.0905 (11)	0.0318 (7)	0.000	0.0003 (5)	0.000
O3	0.0309 (6)	0.0647 (8)	0.0380 (7)	0.000	0.0030 (4)	0.000
N1	0.0342 (7)	0.0434 (8)	0.0281 (7)	0.000	0.0005 (5)	0.000
C1	0.0354 (9)	0.0466 (9)	0.0339 (8)	0.000	−0.0076 (6)	0.000
C2	0.0281 (7)	0.0452 (9)	0.0368 (9)	0.000	−0.0027 (6)	0.000
C3	0.0312 (8)	0.0308 (8)	0.0319 (8)	0.000	−0.0011 (6)	0.000
C4	0.0305 (8)	0.0282 (8)	0.0323 (8)	0.000	−0.0019 (6)	0.000
C5	0.0353 (9)	0.0441 (9)	0.0318 (8)	0.000	−0.0025 (6)	0.000
C6	0.0376 (9)	0.0556 (11)	0.0385 (9)	0.000	−0.0116 (7)	0.000
C7	0.0265 (7)	0.0562 (11)	0.0514 (10)	0.000	−0.0044 (7)	0.000

C8	0.0321 (8)	0.0469 (10)	0.0379 (9)	0.000	0.0034 (6)	0.000
C9	0.0310 (8)	0.0303 (8)	0.0324 (8)	0.000	−0.0031 (6)	0.000
C10	0.0310 (8)	0.0353 (8)	0.0376 (8)	0.000	−0.0003 (6)	0.000
C11	0.0355 (9)	0.0700 (13)	0.0418 (10)	0.000	0.0086 (7)	0.000
C12	0.0352 (11)	0.149 (3)	0.0661 (16)	0.000	0.0053 (10)	0.000
C13	0.0450 (10)	0.0733 (13)	0.0313 (9)	0.000	0.0031 (7)	0.000

Geometric parameters (Å, º)

O1—C1	1.231 (2)	C6—H6	0.9300
O2—C10	1.200 (2)	C6—C7	1.381 (3)
O3—C10	1.3312 (19)	C7—H7	0.9300
O3—C11	1.454 (2)	C7—C8	1.371 (2)
N1—C1	1.372 (2)	C8—H8	0.9300
N1—C9	1.3952 (19)	C8—C9	1.403 (2)
N1—C13	1.469 (2)	C11—H11A	0.9700
C1—C2	1.450 (2)	C11—H11B	0.9700
C2—H2	0.9300	C11—C12	1.490 (3)
C2—C3	1.347 (2)	C12—H12A	0.9600
C3—C4	1.454 (2)	C12—H12B	0.9600
C3—C10	1.505 (2)	C12—H12C	0.9600
C4—C5	1.407 (2)	C13—H13A	0.9600
C4—C9	1.417 (2)	C13—H13B	0.9600
C5—H5	0.9300	C13—H13C	0.9600
C5—C6	1.381 (2)		
C10—O3—C11	116.42 (13)	C7—C8—C9	120.08 (15)
C1—N1—C9	122.79 (13)	C9—C8—H8	120.0
C1—N1—C13	117.13 (13)	N1—C9—C4	120.60 (14)
C9—N1—C13	120.08 (14)	N1—C9—C8	119.52 (13)
O1—C1—N1	121.81 (15)	C8—C9—C4	119.88 (13)
O1—C1—C2	122.00 (15)	O2—C10—O3	122.91 (15)
N1—C1—C2	116.19 (14)	O2—C10—C3	125.97 (14)
C1—C2—H2	118.2	O3—C10—C3	111.12 (13)
C3—C2—C1	123.52 (15)	O3—C11—H11A	110.4
C3—C2—H2	118.2	O3—C11—H11B	110.4
C2—C3—C4	119.33 (14)	O3—C11—C12	106.58 (17)
C2—C3—C10	118.12 (14)	H11A—C11—H11B	108.6
C4—C3—C10	122.55 (13)	C12—C11—H11A	110.4
C5—C4—C3	124.43 (14)	C12—C11—H11B	110.4
C5—C4—C9	118.00 (14)	C11—C12—H12A	109.5
C9—C4—C3	117.57 (13)	C11—C12—H12B	109.5
C4—C5—H5	119.4	C11—C12—H12C	109.5
C6—C5—C4	121.12 (15)	H12A—C12—H12B	109.5
C6—C5—H5	119.4	H12A—C12—H12C	109.5
C5—C6—H6	120.1	H12B—C12—H12C	109.5
C7—C6—C5	119.86 (15)	N1—C13—H13A	109.5
C7—C6—H6	120.1	N1—C13—H13B	109.5

C6—C7—H7	119.5	N1—C13—H13C	109.5
C8—C7—C6	121.05 (16)	H13A—C13—H13B	109.5
C8—C7—H7	119.5	H13A—C13—H13C	109.5
C7—C8—H8	120.0	H13B—C13—H13C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O2	0.93	2.24	2.892 (2)	126
C8—H8···O1^{i}	0.93	2.37	3.285 (2)	168

Symmetry code: (i) $x+1/2, -y+1/2, -z+1/2$.