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Ethyl 6-chloro-2-oxo-4-phenyl-1,2-dihydroquinoline-3-carboxylate

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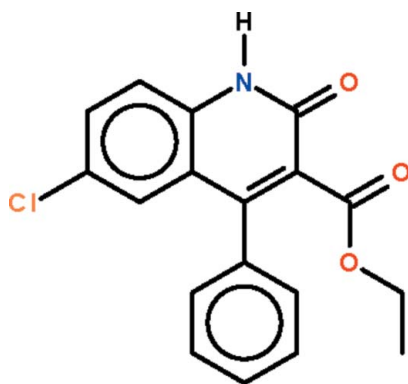
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 Key indicators: single-crystal X-ray study; $T = 290$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.046; wR factor = 0.129; data-to-parameter ratio = 17.4.

In the title compound, $\text{C}_{18}\text{H}_{14}\text{ClNO}_3$, the dihydroquinolin-2-one ring system is almost planar (r.m.s. deviation = 0.033 Å). The carboxylate plane and the phenyl group are twisted away from the dihydroquinolin-2-one ring system by 50.3 (1) and 64.9 (1)°, respectively. In the crystal structure, inversion-related molecules form $R_2^2(8)$ dimers *via* pairs of $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For crystal structures of related compounds, see: Baumer *et al.* (2001); Subashini *et al.* (2009).



Experimental

Crystal data

 $\text{C}_{18}\text{H}_{14}\text{ClNO}_3$
 $M_r = 327.75$

 Monoclinic, $P2_1/c$
 $a = 10.176$ (1) Å
 $b = 15.629$ (2) Å
 $c = 11.282$ (1) Å
 $\beta = 115.463$ (1)°
 $V = 1619.9$ (3) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.25$ mm⁻¹
 $T = 290$ K
 $0.35 \times 0.31 \times 0.23$ mm

Data collection

 Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.918$, $T_{\max} = 0.945$

 13600 measured reflections
 3699 independent reflections
 2906 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.129$
 $S = 1.00$
 3699 reflections
 213 parameters

 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.23$ e Å⁻³
 $\Delta\rho_{\min} = -0.20$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}\cdots\text{O1}^i$	0.88 (2)	1.89 (2)	2.763 (2)	178 (2)

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

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2958).

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supporting information

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Ethyl 6-chloro-2-oxo-4-phenyl-1,2-dihydroquinoline-3-carboxylate

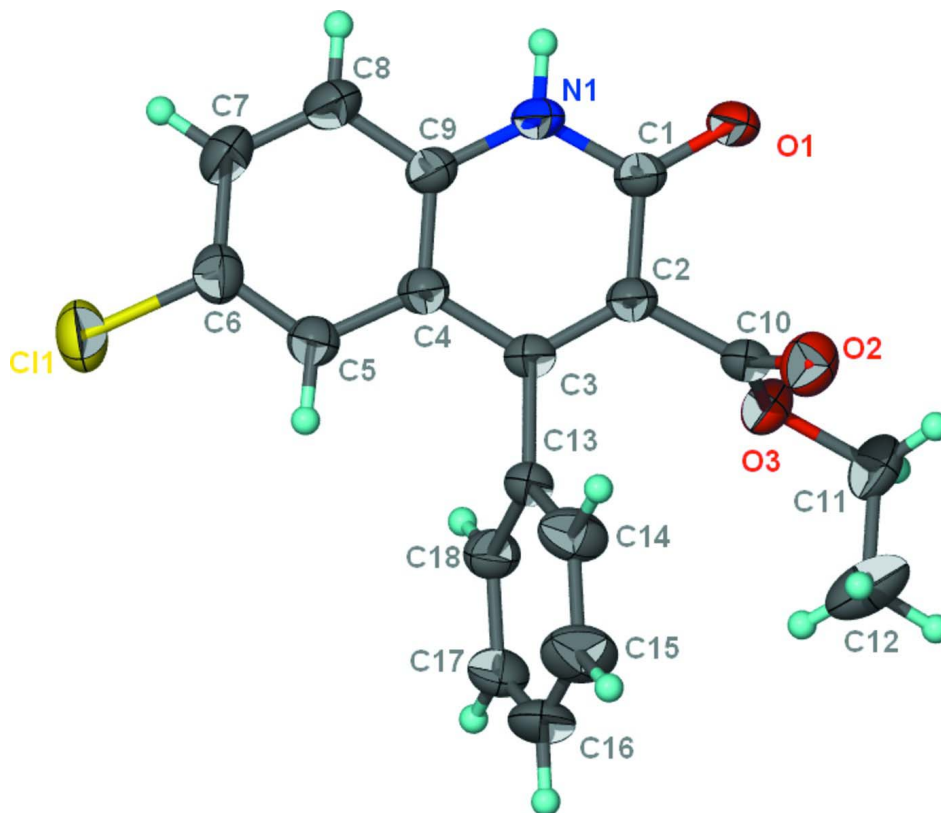
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S1. Experimental

(2-Amino-5-chlorophenyl)(phenyl)methanone (1 mmol) and diethyl malonate (1.2 mmol) along with a catalytic amount of piperidine were heated at 453 K; the reaction was monitored by TLC. After completion, the reaction mixture was poured into the water. The organic product was extracted with ethyl acetate. The crude product was then purified by silica-gel column chromatography, with petroleum ether and ethyl acetate as eluant. Single crystals were obtained by recrystallization from ethyl acetate.

S2. Refinement

C-bound H-atoms were placed in calculated positions (C-H = 0.93–0.97 Å) and were included in the refinement in the riding model approximation, with $U_{iso}(\text{H})$ set to $1.2U_{eq}(\text{C})$. The amino H-atom was located in a difference Fourier map, and was freely refined without any restraint.

**Figure 1**

Displacement ellipsoid plot (Barbour, 2001) of $C_{18}H_{14}ClNO_3$ at the 50% probability level. H atoms are drawn as spheres of arbitrary radius.

Ethyl 6-chloro-2-oxo-4-phenyl-1,2-dihydroquinoline-3-carboxylate

Crystal data

$C_{18}H_{14}ClNO_3$

$M_r = 327.75$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 10.176 (1) \text{ \AA}$

$b = 15.629 (2) \text{ \AA}$

$c = 11.282 (1) \text{ \AA}$

$\beta = 115.463 (1)^\circ$

$V = 1619.9 (3) \text{ \AA}^3$

$Z = 4$

$F(000) = 680$

$D_x = 1.344 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1123 reflections

$\theta = 2.9\text{--}20.7^\circ$

$\mu = 0.25 \text{ mm}^{-1}$

$T = 290 \text{ K}$

Block, colourless

$0.35 \times 0.31 \times 0.23 \text{ mm}$

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.918$, $T_{\max} = 0.945$

13600 measured reflections

3699 independent reflections

2906 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.022$

$\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 2.2^\circ$

$h = -13 \rightarrow 13$

$k = -20 \rightarrow 19$

$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.129$
 $S = 1.00$
 3699 reflections
 213 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0664P)^2 + 0.4015P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.23 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.20 \text{ e } \text{Å}^{-3}$

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.54343 (6)	0.02895 (3)	0.68514 (6)	0.0743 (2)
N1	0.46268 (16)	0.38491 (9)	0.50054 (15)	0.0526 (4)
H1	0.527 (2)	0.4235 (14)	0.546 (2)	0.066 (6)*
O1	0.33674 (13)	0.49099 (7)	0.36051 (13)	0.0569 (3)
O2	0.13633 (14)	0.40486 (9)	0.08985 (13)	0.0605 (4)
O3	0.00732 (12)	0.38779 (8)	0.20662 (11)	0.0487 (3)
C1	0.35167 (17)	0.41406 (10)	0.38896 (17)	0.0454 (4)
C2	0.25139 (17)	0.34892 (10)	0.30798 (15)	0.0404 (3)
C3	0.26581 (16)	0.26477 (10)	0.34062 (15)	0.0384 (3)
C4	0.38454 (16)	0.23795 (10)	0.46323 (16)	0.0405 (4)
C5	0.40613 (18)	0.15305 (11)	0.50898 (16)	0.0450 (4)
H5	0.3452	0.1098	0.4580	0.054*
C6	0.5166 (2)	0.13411 (12)	0.62836 (19)	0.0530 (4)
C7	0.6110 (2)	0.19671 (14)	0.7067 (2)	0.0648 (5)
H7	0.6853	0.1826	0.7880	0.078*
C8	0.5932 (2)	0.27920 (13)	0.6629 (2)	0.0634 (5)
H8	0.6566	0.3213	0.7144	0.076*
C9	0.48116 (18)	0.30096 (11)	0.54190 (17)	0.0465 (4)
C10	0.12728 (17)	0.38317 (10)	0.18746 (15)	0.0411 (4)
C11	-0.1227 (2)	0.42135 (14)	0.09932 (19)	0.0620 (5)
H11A	-0.0961	0.4681	0.0573	0.074*
H11B	-0.1882	0.4437	0.1338	0.074*
C12	-0.1985 (2)	0.3536 (2)	0.0004 (2)	0.0894 (8)
H12A	-0.2907	0.3749	-0.0625	0.134*
H12B	-0.2140	0.3044	0.0438	0.134*
H12C	-0.1398	0.3378	-0.0437	0.134*
C13	0.15864 (17)	0.20196 (10)	0.25160 (15)	0.0400 (3)
C14	0.1482 (2)	0.18833 (13)	0.12633 (19)	0.0605 (5)

H14	0.2126	0.2156	0.1001	0.073*
C15	0.0423 (3)	0.13422 (15)	0.0401 (2)	0.0782 (7)
H15	0.0358	0.1254	-0.0438	0.094*
C16	-0.0525 (3)	0.09389 (13)	0.0781 (2)	0.0717 (6)
H16	-0.1231	0.0574	0.0202	0.086*
C17	-0.0437 (2)	0.10699 (12)	0.2011 (2)	0.0619 (5)
H17	-0.1087	0.0796	0.2264	0.074*
C18	0.06135 (19)	0.16075 (11)	0.28790 (17)	0.0499 (4)
H18	0.0667	0.1693	0.3714	0.060*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0758 (4)	0.0599 (3)	0.0810 (4)	0.0093 (2)	0.0277 (3)	0.0260 (3)
N1	0.0408 (8)	0.0370 (8)	0.0583 (9)	-0.0040 (6)	0.0007 (7)	-0.0097 (7)
O1	0.0437 (7)	0.0348 (6)	0.0701 (8)	-0.0018 (5)	0.0034 (6)	-0.0062 (6)
O2	0.0600 (8)	0.0724 (9)	0.0500 (7)	0.0001 (7)	0.0246 (6)	0.0108 (6)
O3	0.0396 (6)	0.0591 (7)	0.0424 (6)	0.0039 (5)	0.0127 (5)	0.0059 (5)
C1	0.0350 (8)	0.0384 (9)	0.0528 (9)	-0.0012 (6)	0.0095 (7)	-0.0072 (7)
C2	0.0370 (8)	0.0392 (8)	0.0407 (8)	-0.0041 (6)	0.0126 (7)	-0.0051 (6)
C3	0.0355 (8)	0.0392 (8)	0.0395 (8)	-0.0040 (6)	0.0152 (7)	-0.0057 (6)
C4	0.0362 (8)	0.0400 (8)	0.0427 (8)	-0.0010 (6)	0.0145 (7)	-0.0050 (7)
C5	0.0426 (9)	0.0419 (9)	0.0486 (9)	-0.0011 (7)	0.0178 (7)	-0.0018 (7)
C6	0.0515 (10)	0.0491 (10)	0.0568 (10)	0.0070 (8)	0.0218 (9)	0.0085 (8)
C7	0.0559 (11)	0.0654 (13)	0.0508 (10)	0.0066 (9)	0.0018 (9)	0.0056 (9)
C8	0.0521 (11)	0.0556 (11)	0.0571 (11)	-0.0020 (9)	-0.0007 (9)	-0.0076 (9)
C9	0.0382 (8)	0.0423 (9)	0.0477 (9)	0.0008 (7)	0.0079 (7)	-0.0049 (7)
C10	0.0413 (8)	0.0349 (8)	0.0405 (8)	-0.0062 (6)	0.0115 (7)	-0.0048 (6)
C11	0.0442 (10)	0.0760 (14)	0.0559 (11)	0.0132 (9)	0.0121 (9)	0.0111 (10)
C12	0.0518 (13)	0.133 (2)	0.0645 (14)	-0.0043 (14)	0.0072 (11)	-0.0217 (15)
C13	0.0406 (8)	0.0347 (8)	0.0398 (8)	-0.0046 (6)	0.0127 (7)	-0.0029 (6)
C14	0.0770 (13)	0.0589 (12)	0.0531 (10)	-0.0188 (10)	0.0352 (10)	-0.0146 (9)
C15	0.1034 (18)	0.0738 (15)	0.0521 (12)	-0.0204 (13)	0.0283 (12)	-0.0255 (10)
C16	0.0731 (14)	0.0518 (12)	0.0663 (13)	-0.0210 (10)	0.0073 (11)	-0.0175 (10)
C17	0.0506 (11)	0.0514 (11)	0.0736 (13)	-0.0162 (8)	0.0171 (10)	0.0005 (9)
C18	0.0511 (10)	0.0508 (10)	0.0450 (9)	-0.0105 (8)	0.0181 (8)	-0.0008 (7)

Geometric parameters (Å, °)

C11—C6	1.7424 (19)	C8—C9	1.393 (2)
N1—C1	1.359 (2)	C8—H8	0.93
N1—C9	1.378 (2)	C11—C12	1.490 (3)
N1—H1	0.88 (2)	C11—H11A	0.97
O1—C1	1.237 (2)	C11—H11B	0.97
O2—C10	1.193 (2)	C12—H12A	0.96
O3—C10	1.330 (2)	C12—H12B	0.96
O3—C11	1.454 (2)	C12—H12C	0.96
C1—C2	1.453 (2)	C13—C18	1.382 (2)

C2—C3	1.357 (2)	C13—C14	1.388 (2)
C2—C10	1.501 (2)	C14—C15	1.386 (3)
C3—C4	1.453 (2)	C14—H14	0.93
C3—C13	1.490 (2)	C15—C16	1.367 (3)
C4—C9	1.405 (2)	C15—H15	0.93
C4—C5	1.406 (2)	C16—C17	1.367 (3)
C5—C6	1.365 (2)	C16—H16	0.93
C5—H5	0.93	C17—C18	1.381 (3)
C6—C7	1.389 (3)	C17—H17	0.93
C7—C8	1.365 (3)	C18—H18	0.93
C7—H7	0.93		
C1—N1—C9	124.83 (14)	O2—C10—C2	124.65 (15)
C1—N1—H1	115.4 (14)	O3—C10—C2	110.23 (13)
C9—N1—H1	119.8 (14)	O3—C11—C12	111.10 (18)
C10—O3—C11	117.05 (13)	O3—C11—H11A	109.4
O1—C1—N1	121.80 (15)	C12—C11—H11A	109.4
O1—C1—C2	122.91 (15)	O3—C11—H11B	109.4
N1—C1—C2	115.28 (15)	C12—C11—H11B	109.4
C3—C2—C1	122.95 (15)	H11A—C11—H11B	108.0
C3—C2—C10	122.83 (14)	C11—C12—H12A	109.5
C1—C2—C10	114.18 (14)	C11—C12—H12B	109.5
C2—C3—C4	119.12 (14)	H12A—C12—H12B	109.5
C2—C3—C13	119.56 (14)	C11—C12—H12C	109.5
C4—C3—C13	121.31 (14)	H12A—C12—H12C	109.5
C9—C4—C5	118.23 (15)	H12B—C12—H12C	109.5
C9—C4—C3	117.95 (14)	C18—C13—C14	118.65 (15)
C5—C4—C3	123.80 (14)	C18—C13—C3	121.14 (14)
C6—C5—C4	119.94 (16)	C14—C13—C3	120.06 (14)
C6—C5—H5	120.0	C15—C14—C13	120.29 (18)
C4—C5—H5	120.0	C15—C14—H14	119.9
C5—C6—C7	121.72 (17)	C13—C14—H14	119.9
C5—C6—C11	119.97 (15)	C16—C15—C14	120.11 (19)
C7—C6—C11	118.31 (15)	C16—C15—H15	119.9
C8—C7—C6	119.16 (18)	C14—C15—H15	119.9
C8—C7—H7	120.4	C15—C16—C17	120.18 (18)
C6—C7—H7	120.4	C15—C16—H16	119.9
C7—C8—C9	120.70 (18)	C17—C16—H16	119.9
C7—C8—H8	119.6	C16—C17—C18	120.24 (19)
C9—C8—H8	119.6	C16—C17—H17	119.9
N1—C9—C8	119.95 (16)	C18—C17—H17	119.9
N1—C9—C4	119.80 (15)	C17—C18—C13	120.53 (17)
C8—C9—C4	120.24 (16)	C17—C18—H18	119.7
O2—C10—O3	125.10 (15)	C13—C18—H18	119.7
C9—N1—C1—O1	176.16 (17)	C7—C8—C9—C4	0.1 (3)
C9—N1—C1—C2	-2.6 (3)	C5—C4—C9—N1	-179.57 (16)
O1—C1—C2—C3	-178.45 (16)	C3—C4—C9—N1	-1.1 (2)

N1—C1—C2—C3	0.3 (2)	C5—C4—C9—C8	-1.2 (3)
O1—C1—C2—C10	-0.6 (2)	C3—C4—C9—C8	177.35 (16)
N1—C1—C2—C10	178.23 (15)	C11—O3—C10—O2	0.5 (2)
C1—C2—C3—C4	1.4 (2)	C11—O3—C10—C2	178.93 (14)
C10—C2—C3—C4	-176.30 (14)	C3—C2—C10—O2	-104.7 (2)
C1—C2—C3—C13	-179.62 (14)	C1—C2—C10—O2	77.4 (2)
C10—C2—C3—C13	2.7 (2)	C3—C2—C10—O3	76.87 (19)
C2—C3—C4—C9	-1.0 (2)	C1—C2—C10—O3	-101.02 (16)
C13—C3—C4—C9	-179.99 (14)	C10—O3—C11—C12	82.5 (2)
C2—C3—C4—C5	177.38 (15)	C2—C3—C13—C18	-111.20 (19)
C13—C3—C4—C5	-1.6 (2)	C4—C3—C13—C18	67.7 (2)
C9—C4—C5—C6	1.5 (2)	C2—C3—C13—C14	64.3 (2)
C3—C4—C5—C6	-176.91 (15)	C4—C3—C13—C14	-116.78 (19)
C4—C5—C6—C7	-0.8 (3)	C18—C13—C14—C15	-0.2 (3)
C4—C5—C6—C11	179.98 (13)	C3—C13—C14—C15	-175.75 (19)
C5—C6—C7—C8	-0.4 (3)	C13—C14—C15—C16	-0.1 (4)
C11—C6—C7—C8	178.89 (17)	C14—C15—C16—C17	0.3 (4)
C6—C7—C8—C9	0.7 (3)	C15—C16—C17—C18	-0.3 (4)
C1—N1—C9—C8	-175.35 (18)	C16—C17—C18—C13	0.1 (3)
C1—N1—C9—C4	3.1 (3)	C14—C13—C18—C17	0.2 (3)
C7—C8—C9—N1	178.5 (2)	C3—C13—C18—C17	175.69 (17)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...O1 ⁱ	0.88 (2)	1.89 (2)	2.763 (2)	178 (2)
C11—H11A...O2 ⁱⁱ	0.97	2.51	3.420 (3)	157
C17—H17...O1 ⁱⁱⁱ	0.93	2.51	3.299 (3)	143
C18—H18...O2 ^{iv}	0.93	2.53	3.317 (2)	142

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x, -y+1, -z$; (iii) $-x, y-1/2, -z+1/2$; (iv) $x, -y+1/2, z+1/2$.