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Prop-2-ynyl 2-oxo-1-(prop-2-ynyl)-1,2-dihydroquinoline-4-carboxylate

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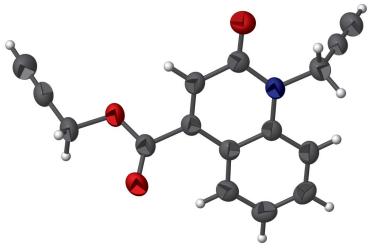
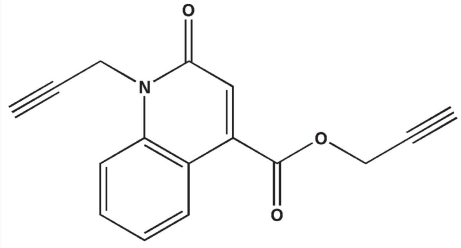
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{16}H_{11}NO_3$, the dihedral angles between the mean planes of the quinolone ring system and the prop-2-ynyl and carboxyprop-2-ynyl groups are 87.9 (8) and 41.6 (8)°, respectively. In the crystal, a weak C—H···O interaction links the molecules into chains along the *c*-axis direction and weak π–π stacking interactions further stabilize the crystal packing.

3D view



Chemical scheme



Structure description

Quinolone derivatives are a classical division of organic chemistry and many of these molecules have shown remarkable biological properties, including exceptional antibacterial activity (Chai *et al.*, 2011; Hoshino *et al.*, 2008). Quinolone derivatives are also frequently associated with medicinal applications, such as anti-fungal (Musiol *et al.*, 2010), anti-tumoral (Bergh *et al.*, 1997) and anti-cancer drugs (Elderfield & LeVon, 1960). As a continuation of our research work devoted to the development of substituted quinoline derivatives (Filali Baba *et al.*, 2017), we report here the synthesis of prop-2-ynyl 1,2-dihydro-2-oxo-1-(prop-2-ynyl)quinoline-4-carboxylate, by reacting 2-oxo-1,2-dihydroquinoline-4-carboxylic acid with 3-bromoprop-1-yne, under phase-transfer catalysis conditions using tetra-*n*-butyl ammonium bromide (TBAB) as a catalyst and potassium carbonate as a base.

The title compound crystallizes with one independent molecule in the asymmetric unit (Fig. 1). The CH_2 group attached to N1 occupies an equatorial position with respect to the mean plane of the quinolone ring. The mean plane through the prop-2-ynyl substituent (N1/C14/C15/C16) makes a dihedral angle of 87.9 (8)° with the mean plane of the

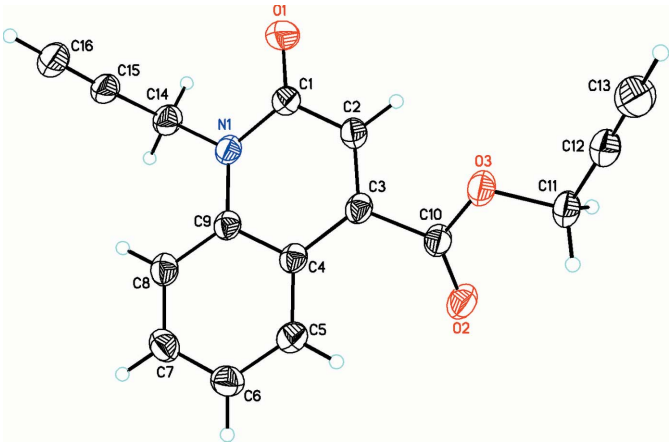


Figure 1
Structure of the title compound, showing the atom-numbering scheme and displacement ellipsoids drawn at the 30% probability level.

quinolone ring system. The dihedral angle between the mean planes of the quinolone ring and the carboxy-prop-2-ynyl unit is 41.6 (8)°.

In the crystal, a single weak C16—H16···O2 intermolecular interaction links the molecules forming one-dimensional chains along the *c* axis (Fig. 2, Table 1). In addition, weak π–π stacking interactions involving the quinolone rings form wave-like layers [intercentroid distances $Cg1\cdots Cg2^{ii}$ = 3.6169 (5) Å, $Cg2\cdots Cg1^{iii}$ = 3.8112 (6) Å; symmetry codes: (ii) $-x, 1-y, 1-z$; (iii) $1-x, 1-y, 1-z$; *Cg*1 and *Cg*2 are the centroids of the N1/C1/C2/C3/C4/C9 and C4–C9 rings, respectively].

Synthesis and crystallization

A solution of 0.5 g (2.64 mmol) 2-oxo-1,2-dihydroquinoline-4-carboxylic acid in 10 ml of DMF was mixed with 0.55 ml (6.34 mmol) 3-bromoprop-1-yne, and 1.09 g (7.92 mmol) K_2CO_3 and 0.17 g (0.52 mmol) TBAB. 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 resulting mixture was chromatographed on a silica gel column [eluent: ethyl acetate / hexane (1/2)]. Crystals were obtained when the solvent was allowed to evaporate (yield = 87%).

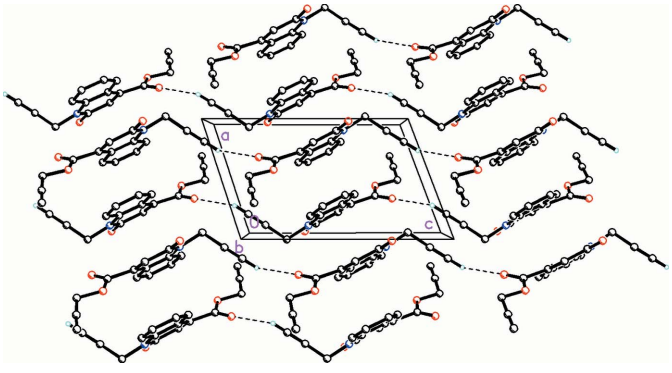


Figure 2
Molecular packing for the title compound, viewed along the *b* axis. Hydrogen bonds are drawn as dashed lines and H atoms not involved in the packing have been omitted for clarity.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16···O2^{i}	0.93	2.34	3.214 (3)	157

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{11}NO_3$
M_r	265.26
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	293
a, *b*, *c* (Å)	7.3070 (11), 8.7763 (13), 11.2927 (13)
α, β, γ (°)	91.059 (11), 107.867 (12), 109.814 (14)
V (Å^3)	642.36 (17)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.79
Crystal size (mm)	0.4 × 0.12 × 0.06
Data collection	
Diffractometer	Rigaku Oxford Diffraction
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku, 2015)
T_{min}, T_{max}	0.833, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	3962, 2418, 1624
R_{int}	0.028
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.613
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.049, 0.142, 1.02
No. of reflections	2418
No. of parameters	181
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.14, −0.20

Computer programs: *CrysAlis PRO* (Rigaku, 2015), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2016* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2017). **2**, x171072 [https://doi.org/10.1107/S2414314617010720]

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Prop-2-ynyl 2-oxo-1-(prop-2-ynyl)-1,2-dihydroquinoline-4-carboxylate

Crystal data

$C_{16}H_{11}NO_3$
M_r = 265.26
Triclinic, $P\overline{1}$
a = 7.3070 (11) Å
b = 8.7763 (13) Å
c = 11.2927 (13) Å
α = 91.059 (11)°
β = 107.867 (12)°
γ = 109.814 (14)°
V = 642.36 (17) Å^3
Z = 2
$F(000)$ = 276
D_x = 1.371 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 943 reflections
θ = 4.1–70.2°
μ = 0.79 mm^{-1}
T = 293 K
Plate, colourless
0.4 × 0.12 × 0.06 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, 2015)
T_{min} = 0.833, T_{max} = 1.000
3962 measured reflections
2418 independent reflections
1624 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 71.0°, θ_{min} = 4.2°
h = −8→6
k = −10→10
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.142
S = 1.02
2418 reflections
181 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0621P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 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.

Refinement. All H atoms were placed in calculated positions and refined using the riding model with C—H bond lengths of 0.93 Å (CH) or 0.97 Å (CH_2). Isotropic displacement parameters for these atoms were set to 1.2 times U_{eq} of the parent atom. In the final cycles of refinement, 4 outliers were omitted.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.0174 (3)	0.0643 (2)	0.29315 (16)	0.0698 (5)
O2	0.3186 (3)	0.4028 (2)	0.79834 (14)	0.0621 (5)
O3	0.4300 (2)	0.2141 (2)	0.74109 (13)	0.0568 (4)
N1	0.0822 (3)	0.3367 (2)	0.32712 (15)	0.0455 (4)
C1	0.0936 (3)	0.1917 (3)	0.3668 (2)	0.0499 (5)
C2	0.1935 (3)	0.1990 (3)	0.5004 (2)	0.0497 (5)
H2	0.2045	0.1041	0.5317	0.060*
C3	0.2707 (3)	0.3380 (3)	0.58080 (18)	0.0433 (5)
C4	0.2700 (3)	0.4906 (3)	0.53617 (18)	0.0418 (5)
C5	0.3604 (3)	0.6417 (3)	0.6127 (2)	0.0513 (5)
H5	0.4252	0.6466	0.6984	0.062*
C6	0.3554 (4)	0.7834 (3)	0.5639 (2)	0.0585 (6)
H6	0.4145	0.8827	0.6163	0.070*
C7	0.2618 (4)	0.7773 (3)	0.4359 (2)	0.0571 (6)
H7	0.2576	0.8728	0.4029	0.068*
C8	0.1753 (3)	0.6319 (3)	0.3576 (2)	0.0507 (5)
H8	0.1163	0.6300	0.2717	0.061*
C9	0.1753 (3)	0.4868 (3)	0.40563 (19)	0.0424 (5)
C10	0.3424 (3)	0.3264 (3)	0.71837 (19)	0.0477 (5)
C11	0.4825 (4)	0.1777 (3)	0.8687 (2)	0.0624 (6)
H11A	0.3585	0.1216	0.8886	0.075*
H11B	0.5623	0.2777	0.9271	0.075*
C12	0.6031 (4)	0.0739 (3)	0.8771 (2)	0.0645 (7)
C13	0.7044 (5)	−0.0037 (4)	0.8812 (3)	0.0873 (10)
H13	0.7860	−0.0661	0.8845	0.105*
C14	−0.0384 (3)	0.3270 (3)	0.19428 (19)	0.0534 (6)
H14A	−0.1455	0.2193	0.1664	0.064*
H14B	−0.1052	0.4066	0.1867	0.064*
C15	0.0878 (4)	0.3570 (3)	0.1123 (2)	0.0546 (6)
C16	0.1826 (5)	0.3752 (4)	0.0445 (2)	0.0720 (7)
H16	0.2586	0.3898	−0.0098	0.086*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0967 (13)	0.0552 (10)	0.0540 (10)	0.0319 (10)	0.0157 (9)	0.0002 (8)
O2	0.0823 (11)	0.0796 (12)	0.0427 (9)	0.0450 (10)	0.0278 (8)	0.0133 (8)
O3	0.0763 (10)	0.0698 (11)	0.0410 (8)	0.0430 (9)	0.0235 (7)	0.0188 (7)
N1	0.0536 (10)	0.0534 (11)	0.0363 (9)	0.0261 (9)	0.0166 (7)	0.0099 (8)
C1	0.0610 (13)	0.0505 (13)	0.0457 (12)	0.0254 (11)	0.0217 (10)	0.0091 (10)
C2	0.0652 (13)	0.0503 (12)	0.0453 (12)	0.0298 (11)	0.0241 (10)	0.0160 (9)

C3	0.0476 (11)	0.0541 (12)	0.0395 (11)	0.0259 (10)	0.0212 (9)	0.0114 (9)
C4	0.0429 (10)	0.0502 (12)	0.0413 (11)	0.0217 (9)	0.0208 (8)	0.0086 (9)
C5	0.0569 (12)	0.0546 (13)	0.0465 (12)	0.0216 (11)	0.0211 (10)	0.0054 (10)
C6	0.0689 (14)	0.0478 (13)	0.0650 (15)	0.0197 (12)	0.0323 (12)	0.0053 (11)
C7	0.0688 (14)	0.0494 (13)	0.0683 (15)	0.0288 (12)	0.0347 (12)	0.0190 (11)
C8	0.0560 (12)	0.0581 (14)	0.0479 (12)	0.0286 (11)	0.0216 (10)	0.0166 (10)
C9	0.0449 (10)	0.0494 (12)	0.0428 (11)	0.0232 (9)	0.0211 (8)	0.0107 (9)
C10	0.0551 (12)	0.0552 (13)	0.0405 (11)	0.0254 (11)	0.0199 (9)	0.0131 (9)
C11	0.0902 (17)	0.0701 (16)	0.0387 (12)	0.0441 (14)	0.0198 (12)	0.0188 (11)
C12	0.0861 (17)	0.0691 (16)	0.0412 (12)	0.0391 (15)	0.0124 (12)	0.0127 (11)
C13	0.114 (2)	0.101 (2)	0.0633 (18)	0.073 (2)	0.0142 (16)	0.0096 (16)
C14	0.0577 (13)	0.0604 (14)	0.0403 (11)	0.0254 (11)	0.0095 (10)	0.0063 (10)
C15	0.0749 (14)	0.0583 (14)	0.0374 (11)	0.0346 (12)	0.0161 (10)	0.0088 (10)
C16	0.105 (2)	0.0784 (19)	0.0522 (15)	0.0481 (17)	0.0356 (15)	0.0166 (13)

Geometric parameters (Å, º)

O1—C1	1.229 (3)	C6—H6	0.9300
O2—C10	1.203 (2)	C6—C7	1.387 (3)
O3—C10	1.336 (3)	C7—H7	0.9300
O3—C11	1.444 (2)	C7—C8	1.371 (3)
N1—C1	1.378 (3)	C8—H8	0.9300
N1—C9	1.401 (3)	C8—C9	1.393 (3)
N1—C14	1.472 (3)	C11—H11A	0.9700
C1—C2	1.450 (3)	C11—H11B	0.9700
C2—H2	0.9300	C11—C12	1.454 (3)
C2—C3	1.345 (3)	C12—C13	1.155 (4)
C3—C4	1.441 (3)	C13—H13	0.9300
C3—C10	1.497 (3)	C14—H14A	0.9700
C4—C5	1.399 (3)	C14—H14B	0.9700
C4—C9	1.418 (3)	C14—C15	1.464 (3)
C5—H5	0.9300	C15—C16	1.161 (3)
C5—C6	1.377 (3)	C16—H16	0.9300
C10—O3—C11	115.86 (16)	C7—C8—H8	119.8
C1—N1—C9	123.77 (18)	C7—C8—C9	120.5 (2)
C1—N1—C14	116.01 (19)	C9—C8—H8	119.8
C9—N1—C14	120.21 (18)	N1—C9—C4	119.30 (19)
O1—C1—N1	121.4 (2)	C8—C9—N1	121.09 (19)
O1—C1—C2	122.9 (2)	C8—C9—C4	119.6 (2)
N1—C1—C2	115.6 (2)	O2—C10—O3	123.80 (19)
C1—C2—H2	118.8	O2—C10—C3	125.2 (2)
C3—C2—C1	122.5 (2)	O3—C10—C3	110.91 (17)
C3—C2—H2	118.8	O3—C11—H11A	110.4
C2—C3—C4	120.96 (19)	O3—C11—H11B	110.4
C2—C3—C10	117.7 (2)	O3—C11—C12	106.68 (18)
C4—C3—C10	121.16 (19)	H11A—C11—H11B	108.6
C5—C4—C3	124.25 (19)	C12—C11—H11A	110.4

C5—C4—C9	118.2 (2)	C12—C11—H11B	110.4
C9—C4—C3	117.50 (19)	C13—C12—C11	176.9 (3)
C4—C5—H5	119.3	C12—C13—H13	180.0
C6—C5—C4	121.3 (2)	N1—C14—H14A	109.0
C6—C5—H5	119.3	N1—C14—H14B	109.0
C5—C6—H6	120.2	H14A—C14—H14B	107.8
C5—C6—C7	119.6 (2)	C15—C14—N1	112.87 (18)
C7—C6—H6	120.2	C15—C14—H14A	109.0
C6—C7—H7	119.6	C15—C14—H14B	109.0
C8—C7—C6	120.7 (2)	C16—C15—C14	177.4 (3)
C8—C7—H7	119.6	C15—C16—H16	180.0

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16···O2^{i}	0.93	2.34	3.214 (3)	157

Symmetry code: (i) *x*, *y*, *z*−1.