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1-Ethyl-3-nitroquinolin-4(1*H*)-one

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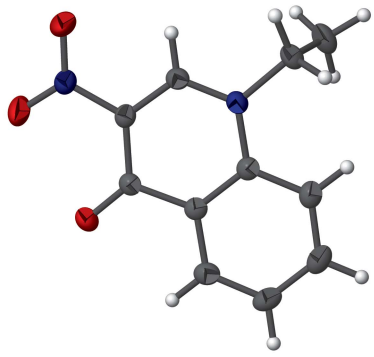
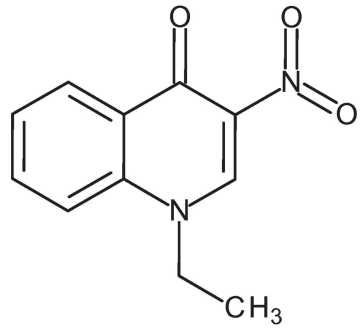
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{11}H_{10}N_2O_3$, was obtained as side-product in a project focussing on the synthesis of carbolines. It was prepared from nitroquinolinone, ethanol and phosphoryl chloride. With the exception of the methyl group [C—N—C—C_{methyl} torsion angle = −96.4 (2)°], the molecule is essentially planar (r.m.s. deviation = 0.033 Å). In the molecular packing, undulating ribbons along the *b* axis are connected *via* C—H···O hydrogen bonds; an intramolecular C—H···O interaction is also noted.

3D view



Chemical scheme



Structure description

The title compound, $C_{11}H_{10}N_2O_3$, Fig. 1, was obtained as side-product in a project focussing on the synthesis of carbolines (Dassonneville *et al.*, 2011; Letessier & Detert, 2012; Letessier *et al.*, 2013) and larger heterocycles with azolo-azine fragments (Glang *et al.*, 2014; Rieth *et al.*, 2014). This compound is one of a series of unexpected side-products in quinoline chemistry (Geffe *et al.*, 2012). The chlorination of nitroquinolone (Bachman *et al.*, 1947) containing ethanol according to Van Galen (Van Galen *et al.*, 1991) yielded the *N*-ethyl quinolone. A large number of quinolones are used in both human and veterinary medicine (Milata *et al.*, 2000). They possess a wide range of biological activities ranging from antibiotic to anticarcinogenic. A carboxyl group in position 3 with a carbonyl group in the 4-position plays an important role in the interaction of a quinoline with DNA-gyrase, the oxo-form can be stabilized by *N*-alkylation (Langer *et al.*, 2011).

The bicyclic ring system is essentially planar, with the exception of the methyl group, with deviations of 0.03 (2) Å. Similarly, the dihedral angle between the ring and the nitro group is only 4.0 (2)°, being stabilized by an intramolecular hydrogen bond (C2—H2···O13; Table 1). Only the methyl group stands nearly orthogonal to the mean plane: the torsion angle C10—C9—N1—C2 amounts to −96.4 (2)°; it acts as a spacer between the molecules. The carbonyl oxygen is the acceptor of a hydrogen bond from C9 (Table 1) whereas the nitro group acts as acceptor for three intermolecular hydrogen bonds

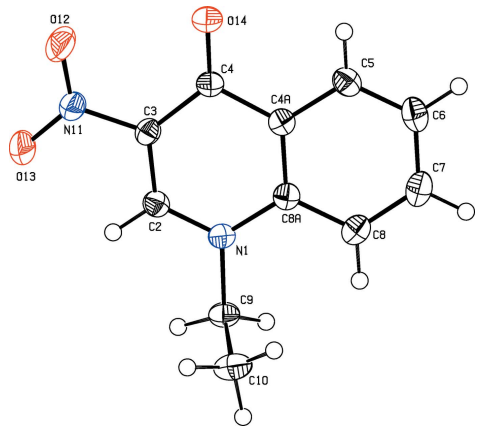


Figure 1
Molecular structure of the title compound with atom labelling and displacement ellipsoids drawn at the 50% probability level.

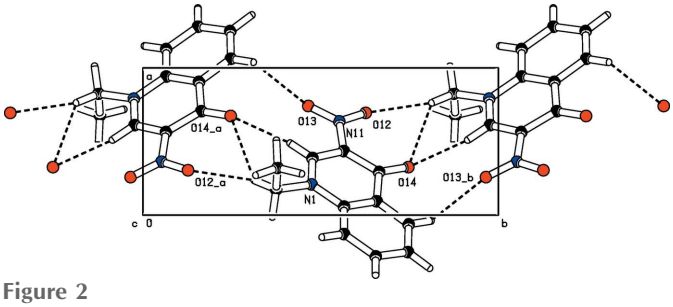


Figure 2
Part of the molecular packing in a view along the *c* axis. Hydrogen bonds are shown as dashed lines.

(Table 1). The molecules are arranged in undulating ribbons along the *b* axis, connected *via* C—H···O hydrogen bonds, Fig. 2.

Synthesis and crystallization

The title compound was prepared from freshly recrystallized ethanol containing 4-hydroxy-3-nitroquinoline (2.28 g) (Bachman *et al.*, 1947), phosphorous pentachloride (2.29 g) and phosphoryl chloride (50 ml) (Van Galen *et al.*, 1991). The mixture was heated to reflux for 2 h, phosphoryl chloride was distilled off and the residue mixed with toluene (20 mL) and added to a stirred ice–water mixture. The organic layer was separated, washed with water, dried, filtered and the product crystallized within 3 d. Yield: 559 mg (22%) of an orange–red solid with m.p. = 495 K. Single crystals were obtained by slow evaporation of a saturated solution in chloroform.

Refinement

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O13	0.95	2.28	2.636 (3)	102
C2—H2···O14^{i}	0.95	2.34	3.239 (2)	159
C9—H9*B*···O14^{i}	0.99	2.57	3.364 (3)	137
C9—H9*B*···O12^{i}	0.99	2.37	3.181 (3)	139

Symmetry code: (i) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_{10}N_2O_3$
M_r	218.21
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	193
a, *b*, *c* (Å)	5.1565 (6), 12.4881 (9), 15.1083 (12)
V (Å^3)	972.90 (15)
Z	4
Radiation type	Mo *Kα*
μ (mm^{-1})	0.11
Crystal size (mm)	0.49 × 0.30 × 0.20
Data collection	
Diffractometer	Stoe IPDS 2T
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	3974, 2353, 2133
R_{int}	0.019
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.664
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.033, 0.088, 1.05
No. of reflections	2353
No. of parameters	146
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.24
Absolute structure	Flack *x* determined using 819 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	2.3 (7)

Computer programs: *X-AREA* (Stoe & Cie, 2011), *X-RED* (Stoe & Cie, 2011), *SIR2004* (Burla *et al.*, 2005), *SHELXL2014* (Sheldrick, 2015), *PLATON* (Spek, 2009).

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full crystallographic data

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1-Ethyl-3-nitroquinolin-4(1*H*)-one

Crystal data

$C_{11}H_{10}N_2O_3$
M_r = 218.21
Orthorhombic, $P2_12_12_1$
a = 5.1565 (6) Å
b = 12.4881 (9) Å
c = 15.1083 (12) Å
V = 972.90 (15) Å^3
Z = 4
$F(000)$ = 456
D_x = 1.490 Mg m^{-3}
Melting point: 495 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6181 reflections
θ = 2.7–28.3°
μ = 0.11 mm^{-1}
T = 193 K
Needle, yellow
0.49 × 0.30 × 0.20 mm

Data collection

Stoe IPDS 2T diffractometer
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus
Detector resolution: 6.67 pixels mm^{-1}
rotation method scans
3974 measured reflections
2353 independent reflections
2133 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 28.2°, θ_{min} = 2.7°
h = −6→5
k = −16→14
l = −20→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.088
S = 1.05
2353 reflections
146 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0465P)^2 + 0.2026P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Absolute structure: Flack x determined using 819 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 2.3 (7)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.2084 (3)	0.47445 (12)	0.65359 (10)	0.0228 (3)
C2	0.3882 (4)	0.47659 (14)	0.71683 (11)	0.0233 (4)
H2	0.4890	0.4140	0.7265	0.028*
C3	0.4353 (4)	0.56468 (15)	0.76878 (12)	0.0232 (4)
C4	0.2937 (4)	0.66408 (14)	0.75900 (11)	0.0222 (3)
C4A	0.0942 (3)	0.65770 (14)	0.68842 (11)	0.0223 (4)
C5	−0.0628 (4)	0.74724 (16)	0.67303 (13)	0.0274 (4)
H5	−0.0392	0.8100	0.7077	0.033*
C6	−0.2510 (4)	0.74565 (17)	0.60848 (14)	0.0318 (4)
H6	−0.3600	0.8061	0.5998	0.038*
C7	−0.2803 (4)	0.65477 (18)	0.55584 (13)	0.0331 (5)
H7	−0.4070	0.6544	0.5102	0.040*
C8	−0.1285 (4)	0.56560 (17)	0.56906 (12)	0.0282 (4)
H8	−0.1498	0.5043	0.5325	0.034*
C8A	0.0581 (4)	0.56545 (15)	0.63672 (11)	0.0228 (4)
C9	0.1748 (4)	0.37471 (15)	0.60113 (12)	0.0272 (4)
H9A	−0.0119	0.3638	0.5888	0.033*
H9B	0.2368	0.3129	0.6363	0.033*
C10	0.3217 (5)	0.37880 (19)	0.51459 (14)	0.0376 (5)
H10A	0.2707	0.3179	0.4776	0.056*
H10B	0.5084	0.3754	0.5264	0.056*
H10C	0.2811	0.4458	0.4837	0.056*
N11	0.6373 (3)	0.55207 (13)	0.83436 (10)	0.0280 (4)
O12	0.6949 (4)	0.62668 (14)	0.88100 (12)	0.0538 (5)
O13	0.7418 (5)	0.46513 (15)	0.84251 (15)	0.0701 (8)
O14	0.3262 (3)	0.74669 (11)	0.80200 (9)	0.0301 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0253 (7)	0.0216 (7)	0.0214 (7)	−0.0007 (6)	0.0009 (6)	−0.0013 (6)
C2	0.0258 (8)	0.0218 (8)	0.0225 (8)	0.0011 (7)	0.0019 (7)	0.0017 (7)
C3	0.0238 (9)	0.0262 (9)	0.0197 (8)	−0.0009 (7)	−0.0004 (6)	0.0013 (7)
C4	0.0232 (8)	0.0229 (8)	0.0205 (7)	−0.0015 (7)	0.0034 (6)	0.0001 (6)
C4A	0.0217 (8)	0.0236 (8)	0.0216 (8)	−0.0013 (6)	0.0035 (6)	0.0024 (7)
C5	0.0279 (9)	0.0256 (9)	0.0287 (9)	0.0023 (8)	0.0041 (7)	0.0022 (8)
C6	0.0280 (9)	0.0338 (10)	0.0337 (10)	0.0061 (8)	0.0015 (8)	0.0093 (8)
C7	0.0267 (10)	0.0441 (12)	0.0285 (9)	0.0002 (8)	−0.0040 (7)	0.0060 (8)
C8	0.0266 (9)	0.0333 (10)	0.0248 (8)	−0.0039 (8)	−0.0007 (7)	0.0005 (7)
C8A	0.0223 (8)	0.0253 (9)	0.0207 (8)	−0.0012 (7)	0.0031 (6)	0.0022 (7)
C9	0.0304 (9)	0.0222 (8)	0.0290 (8)	−0.0040 (8)	0.0002 (8)	−0.0050 (7)
C10	0.0399 (11)	0.0400 (11)	0.0329 (10)	−0.0059 (10)	0.0053 (9)	−0.0130 (9)
N11	0.0314 (9)	0.0289 (8)	0.0237 (7)	0.0013 (7)	−0.0041 (7)	0.0012 (6)
O12	0.0696 (13)	0.0336 (8)	0.0583 (11)	0.0032 (9)	−0.0404 (10)	−0.0085 (8)
O13	0.0946 (17)	0.0451 (10)	0.0708 (13)	0.0360 (11)	−0.0543 (13)	−0.0200 (9)

O14	0.0344 (7)	0.0247 (6)	0.0313 (7)	−0.0001 (6)	−0.0029 (6)	−0.0056 (6)

Geometric parameters (Å, º)

N1—C2	1.332 (2)	C6—H6	0.9500
N1—C8A	1.399 (2)	C7—C8	1.376 (3)
N1—C9	1.486 (2)	C7—H7	0.9500
C2—C3	1.373 (2)	C8—C8A	1.404 (3)
C2—H2	0.9500	C8—H8	0.9500
C3—N11	1.446 (2)	C9—C10	1.512 (3)
C3—C4	1.448 (2)	C9—H9A	0.9900
C4—O14	1.231 (2)	C9—H9B	0.9900
C4—C4A	1.484 (2)	C10—H10A	0.9800
C4A—C5	1.400 (2)	C10—H10B	0.9800
C4A—C8A	1.404 (2)	C10—H10C	0.9800
C5—C6	1.376 (3)	N11—O12	1.205 (2)
C5—H5	0.9500	N11—O13	1.218 (2)
C6—C7	1.394 (3)		
C2—N1—C8A	120.01 (15)	C6—C7—H7	119.5
C2—N1—C9	118.71 (15)	C7—C8—C8A	119.79 (18)
C8A—N1—C9	121.28 (15)	C7—C8—H8	120.1
N1—C2—C3	123.33 (16)	C8A—C8—H8	120.1
N1—C2—H2	118.3	N1—C8A—C8	120.89 (17)
C3—C2—H2	118.3	N1—C8A—C4A	119.45 (15)
C2—C3—N11	115.59 (16)	C8—C8A—C4A	119.65 (17)
C2—C3—C4	122.64 (16)	N1—C9—C10	112.00 (16)
N11—C3—C4	121.77 (16)	N1—C9—H9A	109.2
O14—C4—C3	126.59 (17)	C10—C9—H9A	109.2
O14—C4—C4A	121.26 (17)	N1—C9—H9B	109.2
C3—C4—C4A	112.15 (15)	C10—C9—H9B	109.2
C5—C4A—C8A	119.09 (16)	H9A—C9—H9B	107.9
C5—C4A—C4	118.52 (16)	C9—C10—H10A	109.5
C8A—C4A—C4	122.39 (16)	C9—C10—H10B	109.5
C6—C5—C4A	120.93 (19)	H10A—C10—H10B	109.5
C6—C5—H5	119.5	C9—C10—H10C	109.5
C4A—C5—H5	119.5	H10A—C10—H10C	109.5
C5—C6—C7	119.53 (19)	H10B—C10—H10C	109.5
C5—C6—H6	120.2	O12—N11—O13	121.38 (17)
C7—C6—H6	120.2	O12—N11—C3	119.60 (16)
C8—C7—C6	120.94 (19)	O13—N11—C3	119.00 (16)
C8—C7—H7	119.5		
C8A—N1—C2—C3	0.7 (3)	C2—N1—C8A—C8	178.58 (17)
C9—N1—C2—C3	179.78 (16)	C9—N1—C8A—C8	−0.4 (2)
N1—C2—C3—N11	179.74 (16)	C2—N1—C8A—C4A	−1.6 (2)
N1—C2—C3—C4	−0.4 (3)	C9—N1—C8A—C4A	179.37 (16)
C2—C3—C4—O14	−179.06 (18)	C7—C8—C8A—N1	177.65 (18)

N11—C3—C4—O14	0.8 (3)	C7—C8—C8A—C4A	−2.2 (3)
C2—C3—C4—C4A	0.9 (2)	C5—C4A—C8A—N1	−177.91 (16)
N11—C3—C4—C4A	−179.29 (15)	C4—C4A—C8A—N1	2.2 (2)
O14—C4—C4A—C5	−1.7 (3)	C5—C4A—C8A—C8	1.9 (2)
C3—C4—C4A—C5	178.35 (16)	C4—C4A—C8A—C8	−177.99 (16)
O14—C4—C4A—C8A	178.16 (17)	C2—N1—C9—C10	−96.4 (2)
C3—C4—C4A—C8A	−1.8 (2)	C8A—N1—C9—C10	82.6 (2)
C8A—C4A—C5—C6	0.2 (3)	C2—C3—N11—O12	178.05 (19)
C4—C4A—C5—C6	−179.94 (17)	C4—C3—N11—O12	−1.8 (3)
C4A—C5—C6—C7	−2.0 (3)	C2—C3—N11—O13	−3.6 (3)
C5—C6—C7—C8	1.7 (3)	C4—C3—N11—O13	176.6 (2)
C6—C7—C8—C8A	0.3 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O13	0.95	2.28	2.636 (3)	102
C2—H2···O14^{i}	0.95	2.34	3.239 (2)	159
C9—H9*B*···O14^{i}	0.99	2.57	3.364 (3)	137
C9—H9*B*···O12^{i}	0.99	2.37	3.181 (3)	139

Symmetry code: (i) −*x*+1, *y*−1/2, −*z*+3/2.