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1-Ethyl-4-hydroxyquinolin-2(1*H*)-one

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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 = 0.103
Data-to-parameter ratio = 8.9

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

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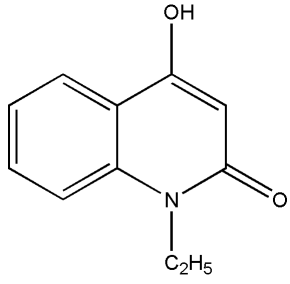
X-ray diffraction study of the title compound, $C_{11}H_{11}NO_2$, (I), has shown that the molecular geometry is similar to that of 1-ethyl-4-hydroxy-2-oxo-1,2-dihydroquinoline-3-carboxylic acid, (II) [Shishkina, Shishkin, Ukrainets, Dakkah & Sidorenko (2002). *Acta Cryst.* E**58**, o254–o256]. Observed elongations of the bonds around the N atom are caused by steric hindrance between the ethyl H atoms and the planar quinolone fragment. Exocyclic angles in the 4-position are also distorted, but, in contrast to (II), this distortion is caused by the formation of zigzag hydrogen-bonded chains along the [010] direction.

Comment

Derivatives of 4-hydroxyquinolin-2-one have been the focus of several structural investigations (Ukrainets *et al.*, 1992, 1996; Garsia Ruano *et al.*, 1991; Shishkina *et al.*, 2002) because they can exist in different tautomeric forms, particularly depending on the presence and nature of the substituent in the 3-position. In the present paper, we report the crystal and molecular structure of 1-ethyl-4-hydroxyquinol-2(1*H*)-one, (I), in which an H atom occupies the 3-position (Fig. 1).



(I)

The rings in the title molecule are coplanar, to within 0.02 Å, with maximum deviations of 0.027 (1) and −0.031 (1) Å for atoms N1 and C2, respectively. For the substituent atoms C9, O1 and O2, the corresponding deviations are 0.018 (2), 0.082 (1) and 0.035 (1) Å. The arrangement around the N atom is planar, despite the observed steric hindrance between the ethyl H atoms and the quinolone fragment [the contacts O1···H9*a* of 2.33 Å and H8···H9*b* of 2.02 Å are shorter than the sums of the van der Waals radii of 2.72 and 2.40 Å, respectively (Bondi, 1964)]. Details of the molecular geometry in (I) are very similar to those observed in the molecule of 1-ethyl-4-hydroxy-2-oxo-1,2-dihydroquinoline-3-carboxylic acid, (II) (Shishkina *et al.*, 2002), which is a precursor in the synthesis of (I) in accordance with procedure of synthesis (Ukrainets *et al.*, 1996). Thus, C3—C4 is

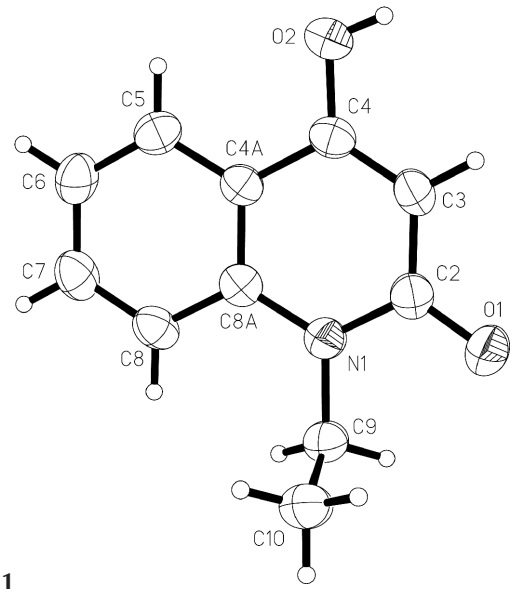


Figure 1
A view of the title molecule, with displacement ellipsoids drawn at the 50% probability level, showing the atom-numbering scheme.

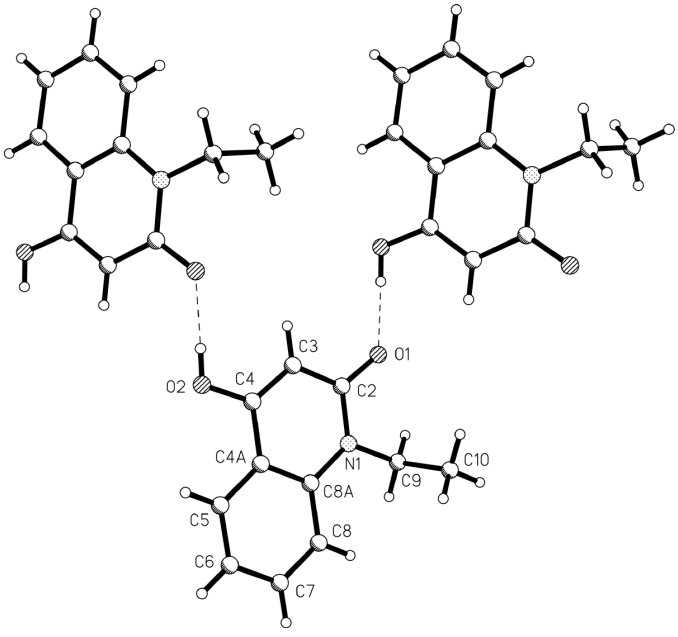


Figure 2
Hydrogen bonds (dashed lines) in the crystal structure of the title compound.

longer than the standard value for a C═C bond (1.334 Å; Bürgi & Dunitz, 1994), and C2—C3 and C4—C4A are shorter than the value of 1.455 Å quoted by Bürgi & Dunitz (1994) for Csp^2—Csp^2 (see Table 1). Futhermore, the N1—C2, N1—C8A and N1—C9 bonds are elongated as a result of the above-mentioned steric hindrance between the methylene H atoms and the quinolone fragment. Distortion of the bond angles around atom C4 is also observed, with the angle O2—C4—C3 noticeably greater than O2—C4—C4A (Table 1). In the structure of (II) these distortions are caused by the presence of the carboxy group in the 3-position and intramolecular O—H···O hydrogen bonding. In the title molecule, the 3-position is not substituted, and the hydrogen bonding is intermolecular (Fig. 2), *via* an O2—H2···O1^{i} hydrogen bond [O2—H2 = 0.82 Å, H2···O1^{i} = 1.76 Å, O2···O1^{i} = 2.580 (2) Å and O2—H2···O1^{i} = 173°; symmetry code: (i) $1-x$, $y-\frac{1}{2}$, $\frac{1}{2}-z$], leading to the formation of infinite zigzag chains along the [010] direction.

Experimental

The preparation procedure for the title compound and the production of crystals for X-ray analysis has been described by Ukrainets *et al.* (1996).

Crystal data

$C_{11}H_{11}NO_2$
M_r = 189.21
Orthorhombic, $P2_12_12_1$
a = 7.8474 (12) Å
b = 8.4660 (14) Å
c = 14.033 (3) Å
V = 932.3 (3) Å^3
Z = 4
D_x = 1.348 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 24 reflections
θ = 11.0–12.0°
μ = 0.09 mm^{-1}
T = 293 (2) K
Polyhedron, colourless
0.35 × 0.30 × 0.30 mm

Data collection

Siemens *P3*/*PC* diffractometer
$2\theta/\theta$ scans
Absorption correction: none
1226 measured reflections
1131 independent reflections
974 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 26.5°
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 17$
2 standard reflections
every 98 reflections
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.103
S = 0.97
1131 reflections
127 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0774P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.262 (2)	C2—C3	1.425 (3)
O2—C4	1.339 (2)	C3—C4	1.355 (3)
N1—C2	1.382 (2)	C4—C4A	1.452 (2)
N1—C8A	1.402 (2)	C4A—C5	1.403 (3)
N1—C9	1.471 (2)	C4A—C8A	1.409 (2)
C2—N1—C8A	121.46 (15)	N1—C2—C3	118.58 (16)
C2—N1—C9	117.17 (15)	C4—C3—C2	121.75 (17)
C8A—N1—C9	121.27 (14)	O2—C4—C3	125.16 (17)
O1—C2—N1	118.20 (16)	O2—C4—C4A	115.04 (16)
O1—C2—C3	123.22 (17)	C3—C4—C4A	119.80 (16)
C8A—N1—C2—O1	176.66 (16)	C9—N1—C2—C3	179.85 (16)
C9—N1—C2—O1	0.3 (2)	C2—C3—C4—O2	−179.71 (17)
C8A—N1—C2—C3	−3.8 (2)	C2—C3—C4—C4A	1.5 (3)

All H atoms were located in a difference map and treated as riding, with an O—H distance of 0.82 Å and C—H distances in the range 0.93–0.97 Å. U_{iso}(H) values were set equal to $1.2U_{eq}$ of the carrier atom ($1.5U_{eq}$ for methyl H atoms).

Data collection: *P*3 (Siemens, 1989); cell refinement: *P*3; data reduction: *XDISK* and *XPREP* (Siemens, 1991); program(s) used to

solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1991); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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