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1-Butyl-4-hydroxy-3-methylquinoline-2(1H)-one

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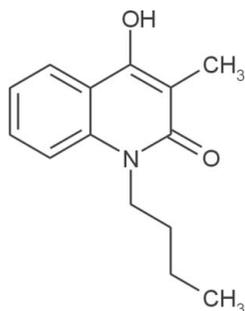
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Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.033; wR factor = 0.086; data-to-parameter ratio = 13.2.

In the crystal of the title compound, $\text{C}_{14}\text{H}_{17}\text{NO}_2$, molecules are arranged into chains along the b axis linked *via* $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds. While the benzene ring is essentially planar, with a maximum deviation from the best plane of 0.003 (1) Å, the pyridine ring is slightly V-shaped: the distance of the carbonyl C atom from the benzene best plane is 0.120 (1) Å. The hydroxy group is inclined markedly towards the benzene ring reducing the $\text{C}-\text{C}-\text{O}$ bond angle to 113.21 (10)°.

Related literature

For the preparation, see: Stadlbauer & Kappe (1985). The title compound is a member of a group of substituted 4-hydroxyquinoline-2-ones used for preparation of new classes of heterocyclic systems, see: Klásek *et al.* (1998); Kafka *et al.* (2002).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{17}\text{NO}_2$
 $M_r = 231.29$
 Monoclinic, $P2_1/c$
 $a = 11.8576$ (7) Å
 $b = 10.7790$ (6) Å
 $c = 9.8835$ (7) Å
 $\beta = 110.749$ (7)°

$V = 1181.31$ (13) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 120$ K
 $0.40 \times 0.40 \times 0.40$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire 2 detector
 Absorption correction: multi-scan (*CrysAlis RED*; Oxford)

Diffraction, 2009
 $T_{\min} = 0.978$, $T_{\max} = 1.000$
 4533 measured reflections
 2077 independent reflections
 1625 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.011$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.086$
 $S = 0.99$
 2077 reflections

157 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.15$ e Å⁻³
 $\Delta\rho_{\min} = -0.21$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O2}-\text{H2A}\cdots\text{O1}^i$	0.84	1.86	2.6529 (14)	156

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2326).

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supporting information

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1-Butyl-4-hydroxy-3-methylquinoline-2(1*H*)-one

Zuzana Kozubková, Marek Nečas and Robert Vícha

S1. Comment

Quinoline derivatives are well known and extensively studied especially for their wide occurrence in nature and for their rich spectrum of biological activities. The title compound is a member of the group of substituted 4-hydroxyquinoline-2-ones used for preparation of new classes of heterocyclic systems (Klásek *et al.*, 1998; Kafka *et al.*, 2002).

The molecule of the title compound (Fig. 1) consists of fused benzene and pyridine rings. The benzene ring is essentially planar with a maximum deviation from the best plane of 0.0026 (12) Å for C6. The pyridine ring is slightly bent along the N1—C3 line with torsion angles C9—N1—C1—C2 and C4—C3—C2—C1 being 5.79 (17) and -6.38 (18)°, respectively. The geometry around C3 markedly differs from the ideal pattern for a *sp*² carbon. All involved atoms C4—C2 and O1 lie in the plane of the phenyl ring (maximum deviation from the best plane is 0.0083 (12) Å for C3) but the valence angles C4—C3—O1 and C2—C3—O1 are 113.21 (10) and 125.91 (11)°. Molecules are linked *via* O2—H2...O1 H-bonds (Fig. 2, Table 1) into chains parallel to the *b*-axis.

S2. Experimental

Title compound was prepared according to a slightly modified procedure published by Stadlbauer & Kappe (1985). A mixture of *N*-butylaniline (16 cm³, 0.1 mol) and diethyl methylmalonate (17.2 cm³, 0.1 mol) was gradually heated in a Wood's metal bath at 413–553 K for 6 h. The reaction was stopped when the amount of condensed ethanol reaches about 93% of the theoretical value. The hot mixture was poured on a metal plate and the crude product was quantitatively transferred into a 500 cm³ Erlenmeyer flask. After addition of 300 cm³ of 0.5 M NaOH and 50 cm³ of toluene the resulting mixture was stirred for 1 h. The suspension was extracted twice with 40 cm³ of toluene and the collected organic portions were treated with powdered activated carbon for 30 min at room temperature. The activated carbon was filtered off and approximately 300–400 cm³ of 5% HCl was added gradually into the filtrate. The precipitated crude product were filtered with suction and washed with water until neutral pH. Single crystals for X-ray analysis were grown by spontaneous evaporation from deuteriochloroform at room temperature.

S3. Refinement

Hydrogen atoms were positioned geometrically and refined as riding using standard *SHELXL-97* facilities, with their *U*_{iso} set to either 1.2*U*_{eq} or 1.5*U*_{eq}(methyl) of their parent atoms.

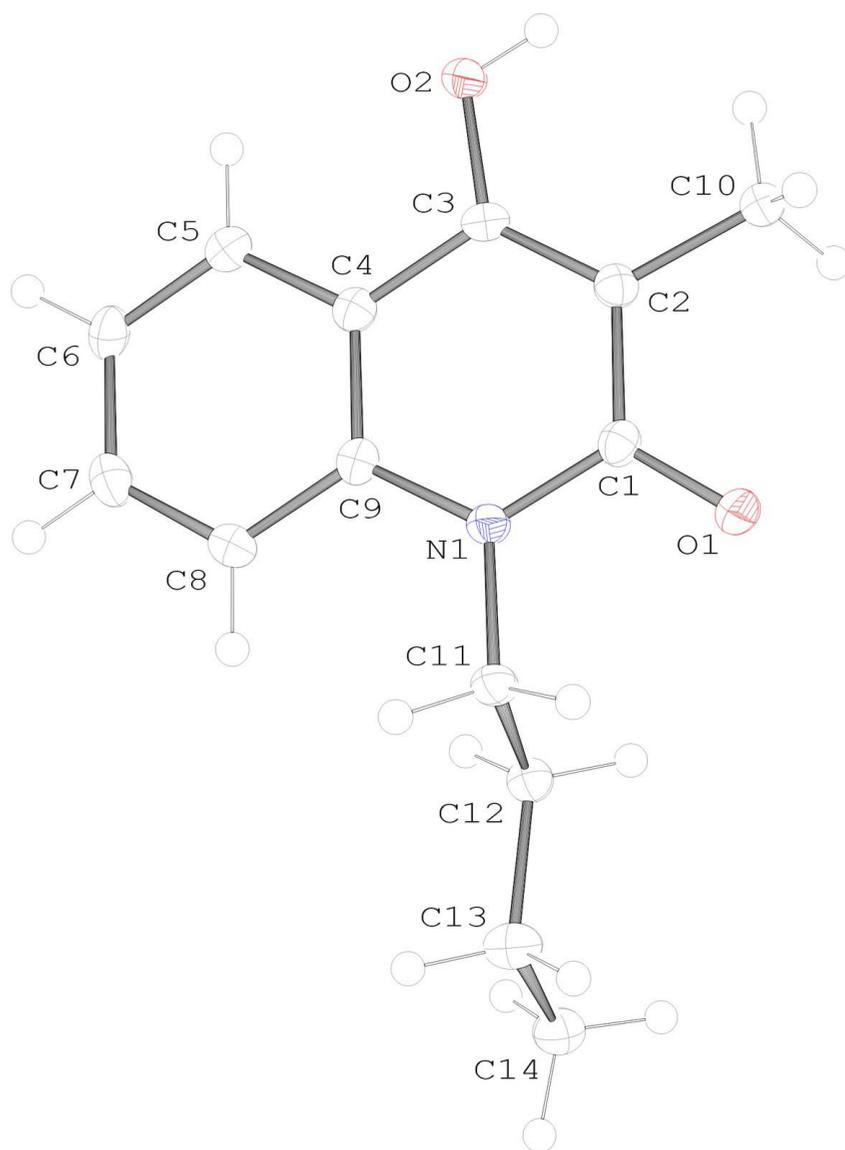


Figure 1

Anisotropic displacement view of the asymmetric unit with atoms represented as 50% probability ellipsoids.

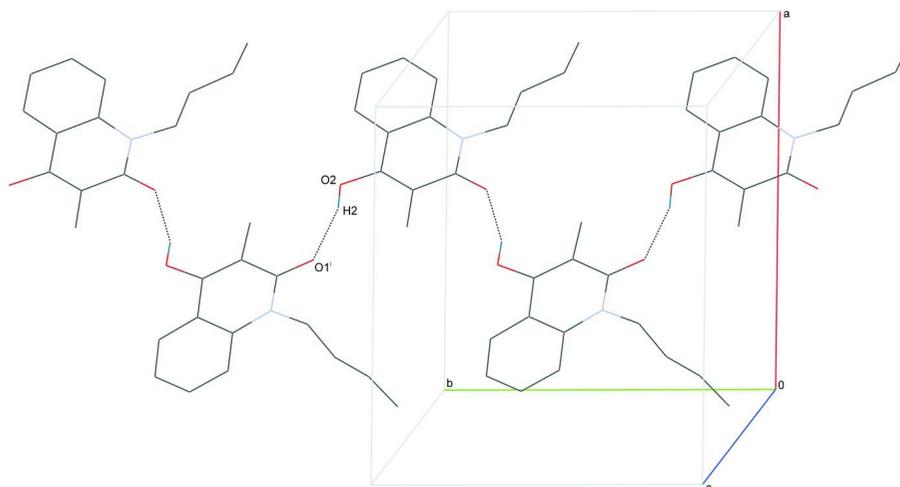


Figure 2

Part of the crystal structure showing chains linked *via* O—H...O hydrogen bonds along the *b*-axis. H-atoms have been omitted except for those participating in H-bonds. Symmetry code: (i) $-x + 1, y + 1/2, -z + 1/2$.

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Crystal data

$C_{14}H_{17}NO_2$
 $M_r = 231.29$
 Monoclinic, $P2_1/c$
 Hall symbol: $-P 2_1/c$
 $a = 11.8576 (7) \text{ \AA}$
 $b = 10.7790 (6) \text{ \AA}$
 $c = 9.8835 (7) \text{ \AA}$
 $\beta = 110.749 (7)^\circ$
 $V = 1181.31 (13) \text{ \AA}^3$
 $Z = 4$

$F(000) = 496$
 $D_x = 1.300 \text{ Mg m}^{-3}$
 Melting point: 471 K
 Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 2482 reflections
 $\theta = 3.0\text{--}27.6^\circ$
 $\mu = 0.09 \text{ mm}^{-1}$
 $T = 120 \text{ K}$
 Block, yellow
 $0.40 \times 0.40 \times 0.40 \text{ mm}$

Data collection

Oxford Diffraction Xcalibur
 diffractometer with a Sapphire2 detector
 Radiation source: Enhance (Mo) X-ray Source
 Graphite monochromator
 Detector resolution: $8.4353 \text{ pixels mm}^{-1}$
 ω scans
 Absorption correction: multi-scan
 (*CrysAlis RED*; Oxford Diffraction, 2009)
 $T_{\min} = 0.978, T_{\max} = 1.000$

4533 measured reflections
 2077 independent reflections
 1625 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.011$
 $\theta_{\max} = 25.0^\circ, \theta_{\min} = 3.0^\circ$
 $h = -13 \rightarrow 14$
 $k = -9 \rightarrow 12$
 $l = -11 \rightarrow 11$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.086$
 $S = 0.99$
 2077 reflections
 157 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-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0533P)^2]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$

$$\Delta\rho_{\max} = 0.15 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.21 \text{ e } \text{\AA}^{-3}$$

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.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.58070 (7)	0.83511 (7)	0.20280 (9)	0.0221 (2)
O2	0.59308 (7)	1.27747 (7)	0.19762 (9)	0.0210 (2)
H2A	0.5408	1.2746	0.2368	0.031*
N1	0.68444 (8)	0.93464 (9)	0.08212 (10)	0.0165 (2)
C1	0.61444 (10)	0.93675 (11)	0.16869 (12)	0.0168 (3)
C2	0.58304 (10)	1.05481 (11)	0.21508 (12)	0.0164 (3)
C3	0.62176 (10)	1.16166 (11)	0.17139 (12)	0.0161 (3)
C4	0.70374 (10)	1.15797 (11)	0.09202 (12)	0.0166 (3)
C5	0.75484 (10)	1.26662 (11)	0.06041 (13)	0.0194 (3)
H5A	0.7342	1.3447	0.0898	0.023*
C6	0.83446 (11)	1.26165 (12)	-0.01253 (13)	0.0220 (3)
H6A	0.8688	1.3357	-0.0330	0.026*
C7	0.86408 (10)	1.14718 (12)	-0.05593 (13)	0.0227 (3)
H7A	0.9185	1.1437	-0.1070	0.027*
C8	0.81590 (10)	1.03880 (11)	-0.02618 (13)	0.0195 (3)
H8A	0.8376	0.9615	-0.0562	0.023*
C9	0.73476 (10)	1.04230 (11)	0.04853 (12)	0.0164 (3)
C10	0.50878 (10)	1.05049 (11)	0.31107 (13)	0.0206 (3)
H10A	0.5068	1.1331	0.3516	0.031*
H10B	0.5448	0.9913	0.3899	0.031*
H10C	0.4264	1.0243	0.2542	0.031*
C11	0.70685 (10)	0.81262 (11)	0.02898 (13)	0.0183 (3)
H11A	0.7145	0.8233	-0.0669	0.022*
H11B	0.6368	0.7580	0.0164	0.022*
C12	0.82009 (10)	0.75039 (12)	0.13077 (13)	0.0198 (3)
H12A	0.8072	0.7273	0.2212	0.024*
H12B	0.8877	0.8103	0.1561	0.024*
C13	0.85459 (11)	0.63463 (12)	0.06562 (14)	0.0251 (3)
H13A	0.7849	0.5773	0.0332	0.030*
H13B	0.8742	0.6585	-0.0203	0.030*
C14	0.96221 (11)	0.56796 (11)	0.17331 (14)	0.0253 (3)
H14A	0.9855	0.4982	0.1252	0.038*

H14B	0.9405	0.5369	0.2540	0.038*
H14C	1.0300	0.6258	0.2101	0.038*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0251 (5)	0.0147 (5)	0.0314 (5)	-0.0013 (4)	0.0161 (4)	0.0010 (4)
O2	0.0232 (5)	0.0150 (5)	0.0303 (5)	0.0007 (4)	0.0164 (4)	-0.0005 (4)
N1	0.0173 (5)	0.0139 (6)	0.0198 (5)	-0.0004 (4)	0.0085 (4)	-0.0015 (4)
C1	0.0153 (6)	0.0165 (7)	0.0178 (6)	-0.0014 (5)	0.0052 (5)	0.0013 (5)
C2	0.0139 (6)	0.0178 (7)	0.0170 (6)	0.0007 (5)	0.0047 (5)	0.0004 (5)
C3	0.0153 (6)	0.0140 (7)	0.0171 (6)	0.0015 (5)	0.0034 (5)	-0.0016 (5)
C4	0.0146 (6)	0.0176 (7)	0.0159 (6)	0.0005 (5)	0.0035 (5)	0.0011 (5)
C5	0.0208 (6)	0.0165 (7)	0.0201 (7)	0.0001 (5)	0.0061 (5)	0.0007 (5)
C6	0.0235 (6)	0.0205 (7)	0.0238 (7)	-0.0053 (5)	0.0107 (5)	0.0026 (6)
C7	0.0213 (6)	0.0279 (8)	0.0219 (7)	-0.0007 (6)	0.0113 (5)	0.0017 (6)
C8	0.0199 (6)	0.0201 (7)	0.0196 (7)	0.0016 (5)	0.0082 (5)	-0.0021 (5)
C9	0.0148 (6)	0.0177 (7)	0.0149 (6)	-0.0013 (5)	0.0032 (5)	0.0011 (5)
C10	0.0241 (6)	0.0156 (7)	0.0262 (7)	0.0004 (5)	0.0138 (5)	0.0002 (5)
C11	0.0207 (6)	0.0144 (7)	0.0216 (7)	-0.0020 (5)	0.0096 (5)	-0.0037 (5)
C12	0.0214 (6)	0.0176 (7)	0.0217 (6)	-0.0006 (5)	0.0093 (5)	-0.0011 (5)
C13	0.0265 (7)	0.0212 (7)	0.0262 (7)	0.0039 (6)	0.0074 (6)	-0.0021 (6)
C14	0.0282 (7)	0.0220 (7)	0.0271 (7)	0.0050 (6)	0.0115 (6)	0.0017 (6)

Geometric parameters (Å, °)

O1—C1	1.2528 (13)	C8—C9	1.4056 (16)
O2—C3	1.3429 (13)	C8—H8A	0.9500
O2—H2A	0.8400	C10—H10A	0.9800
N1—C1	1.3873 (15)	C10—H10B	0.9800
N1—C9	1.3976 (14)	C10—H10C	0.9800
N1—C11	1.4749 (14)	C11—C12	1.5192 (16)
C1—C2	1.4454 (16)	C11—H11A	0.9900
C2—C3	1.3655 (16)	C11—H11B	0.9900
C2—C10	1.5064 (15)	C12—C13	1.5249 (16)
C3—C4	1.4499 (16)	C12—H12A	0.9900
C4—C5	1.4037 (16)	C12—H12B	0.9900
C4—C9	1.4095 (15)	C13—C14	1.5217 (16)
C5—C6	1.3771 (16)	C13—H13A	0.9900
C5—H5A	0.9500	C13—H13B	0.9900
C6—C7	1.3915 (17)	C14—H14A	0.9800
C6—H6A	0.9500	C14—H14B	0.9800
C7—C8	1.3772 (16)	C14—H14C	0.9800
C7—H7A	0.9500		
C3—O2—H2A	109.5	C2—C10—H10A	109.5
C1—N1—C9	122.14 (10)	C2—C10—H10B	109.5
C1—N1—C11	117.19 (9)	H10A—C10—H10B	109.5

C9—N1—C11	120.66 (10)	C2—C10—H10C	109.5
O1—C1—N1	118.00 (10)	H10A—C10—H10C	109.5
O1—C1—C2	122.81 (11)	H10B—C10—H10C	109.5
N1—C1—C2	119.19 (10)	N1—C11—C12	112.73 (9)
C3—C2—C1	119.28 (11)	N1—C11—H11A	109.0
C3—C2—C10	124.21 (11)	C12—C11—H11A	109.0
C1—C2—C10	116.51 (10)	N1—C11—H11B	109.0
O2—C3—C2	125.91 (10)	C12—C11—H11B	109.0
O2—C3—C4	113.21 (10)	H11A—C11—H11B	107.8
C2—C3—C4	120.86 (10)	C11—C12—C13	112.83 (10)
C5—C4—C9	119.36 (11)	C11—C12—H12A	109.0
C5—C4—C3	121.50 (11)	C13—C12—H12A	109.0
C9—C4—C3	119.12 (10)	C11—C12—H12B	109.0
C6—C5—C4	120.94 (12)	C13—C12—H12B	109.0
C6—C5—H5A	119.5	H12A—C12—H12B	107.8
C4—C5—H5A	119.5	C14—C13—C12	112.02 (10)
C5—C6—C7	119.38 (11)	C14—C13—H13A	109.2
C5—C6—H6A	120.3	C12—C13—H13A	109.2
C7—C6—H6A	120.3	C14—C13—H13B	109.2
C8—C7—C6	121.15 (11)	C12—C13—H13B	109.2
C8—C7—H7A	119.4	H13A—C13—H13B	107.9
C6—C7—H7A	119.4	C13—C14—H14A	109.5
C7—C8—C9	120.16 (11)	C13—C14—H14B	109.5
C7—C8—H8A	119.9	H14A—C14—H14B	109.5
C9—C8—H8A	119.9	C13—C14—H14C	109.5
N1—C9—C8	122.13 (11)	H14A—C14—H14C	109.5
N1—C9—C4	118.85 (10)	H14B—C14—H14C	109.5
C8—C9—C4	119.02 (11)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O2—H2A \cdots O1 ⁱ	0.84	1.86	2.6529 (14)	156

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