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Crystal structure of methyl 4-(4-hydroxy-3-nitrophenyl)-2-methyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate, $C_{18}H_{18}N_2O_6$

Table 1: Crystal collection and handling.

Crystal:	Block, colorless
Size:	$0.29 \times 0.25 \times 0.21$ mm
Wavelength:	Mo K_α radiation ($\lambda = 0.71073$ Å)
μ :	0.110 mm^{-1}
Diffractometer, scan mode:	APEX2, Φ and ω -scans
θ_{max} , completeness:	25° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8190, 2896, 0.030
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 2156
$N(\text{param})_{\text{refined}}$:	236
Programs:	Bruker programs [7], SHELX [8], OLEX2 [9]

tions and a list of the atoms including atomic coordinates and displacement parameters.

Comment

4-Arylpolyhydroquinoline derivatives represent one of the most active classes of compounds possessing a wide spectrum of biological activities [1–3]. The interesting biological activity of these 4-arylpolyhydroquinolines made these compounds attractive targets in organic synthesis [4].

In the crystal structure of the title compound (*cf.* the figure), the six-membered ring containing a nitrogen atom is nearly planar and the annulated ring adopts a typical chair conformation. The bond distances and the bond angles in the title compound are comparable with those in reported compounds [5]. The crystal structural features of the title compound are similar to those in 2-methyl-4-(4-nitro-phenyl)-5-oxo-1,4,5,6,7,8-hexahydro-quinoline-3-carboxylic acid ethyl ester [5] and 4-(3,4-dimethyl-phenyl)-2-methyl-5-oxo-1,4,5,6,7,8-hexahydro-quinoline-3-carboxylic acid ethyl ester [6]. Compared with the known molecules, the most differences laid on the functional groups: in 2-methyl-4-(4-nitro-phenyl)-5-oxo-1,4,5,6,7,8-hexahydro-quinoline-3-carboxylic acid ethyl ester, there is one $-C_2H_5$ group at the phenyl moiety. In 4-(3,4-dimethyl-phenyl)-2-methyl-5-oxo-1,4,5,6,7,8-hexahydro-quinoline-3-carboxylic acid ethyl ester, there are two $-CH_3$ groups at the phenyl moiety. In the title molecule the OH and NO_2 substituent at the phenyl moiety form a weak hydrogen bond.

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Abstract

$C_{18}H_{18}N_2O_6$, monoclinic, $P2_1/n$ (no. 14), $a = 7.4809(14)$ Å, $b = 13.082(2)$ Å, $c = 16.870(3)$ Å, $\beta = 93.440(4)^\circ$, $V = 1648.0(5)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0433$, $wR_{\text{ref}}(F^2) = 0.1279$, $T = 296(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement condi-

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.6057(2)	0.82663(12)	−0.05712(10)	0.0479(4)
H1	0.711525	0.815225	−0.072333	0.057*
N2	−0.1011(2)	0.53064(13)	0.10915(9)	0.0478(4)
O1	0.2189(2)	0.92495(11)	0.13334(9)	0.0619(4)
O2	0.5038(2)	0.95085(13)	0.17125(10)	0.0752(5)
O3	−0.2175(2)	0.59437(12)	0.11566(11)	0.0725(5)
O4	−0.1270(2)	0.43930(11)	0.12108(10)	0.0660(5)
O5	0.2108(2)	0.39775(10)	0.11019(9)	0.0581(4)
H5	0.107593	0.381256	0.118097	0.087*
O6	−0.01105(18)	0.79345(11)	−0.10953(8)	0.0536(4)
C1	0.1657(4)	0.97662(19)	0.20270(14)	0.0738(7)
H1A	0.037441	0.977235	0.202824	0.111*
H1B	0.214537	0.941767	0.249174	0.111*
H1C	0.209527	1.045607	0.202711	0.111*
C2	0.3945(3)	0.91688(14)	0.12343(12)	0.0479(5)
C3	0.4264(3)	0.86541(13)	0.04887(11)	0.0411(5)
C4	0.5875(3)	0.86786(13)	0.01733(13)	0.0439(5)
C5	0.7561(3)	0.91366(15)	0.05320(14)	0.0566(6)
H5A	0.815800	0.864920	0.088115	0.085*
H5B	0.728010	0.973704	0.082718	0.085*
H5C	0.832895	0.931831	0.011852	0.085*
C6	0.4621(3)	0.80340(13)	−0.10726(12)	0.0421(5)
C7	0.2963(2)	0.79997(12)	−0.07917(11)	0.0382(4)
C8	0.1413(3)	0.79080(14)	−0.13306(12)	0.0435(5)
C9	0.1691(3)	0.77953(18)	−0.21993(12)	0.0600(6)
H9A	0.163332	0.846427	−0.244747	0.072*
H9B	0.073252	0.738207	−0.244365	0.072*
C10	0.3459(3)	0.73074(17)	−0.23485(13)	0.0610(6)
H10A	0.362922	0.730572	−0.291398	0.073*
H10B	0.346042	0.660380	−0.216704	0.073*
C11	0.4970(3)	0.78801(16)	−0.19201(12)	0.0536(5)
H11A	0.607542	0.750004	−0.195746	0.064*
H11B	0.511467	0.853911	−0.217195	0.064*
C12	0.2722(2)	0.80748(12)	0.00785(11)	0.0390(4)
H12	0.161802	0.845753	0.015236	0.047*
C13	0.2516(2)	0.70124(13)	0.04298(10)	0.0379(4)
C14	0.3974(3)	0.63631(14)	0.05278(13)	0.0500(5)
H14	0.510414	0.660952	0.042758	0.060*
C15	0.3805(3)	0.53733(15)	0.07667(13)	0.0522(5)
H15	0.481812	0.496234	0.082716	0.063*
C16	0.2159(3)	0.49727(13)	0.09199(11)	0.0422(5)
C17	0.0716(2)	0.56397(13)	0.08716(10)	0.0387(4)
C18	0.0899(3)	0.66415(13)	0.06239(11)	0.0398(4)
H18	−0.009798	0.706737	0.058958	0.048*

Experimental

The title compound was synthesized according to a reported procedure. A mixture of 3,5-cyclohexanedione (10 mmol), 3-nitro-4-hydroxybenzaldehyde (10 mmol), 3-amino-2-butenic acid methyl ester (10 mmol) in ethanol (100 mL) was refluxed for 2–3 h and then cooled to room temperature. After filtering the precipitates, they were sequentially washed with ice-cooled water and ethanol and then dried under a vacuum.

Refinement

H atoms bonded to C and N atoms were positioned geometrically and refined using a riding model, with C–H = 0.96 Å and N–H = 0.86 Å with $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{eq}}(\text{C})$ and 1.2 times $U_{\text{eq}}(\text{N})$.

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