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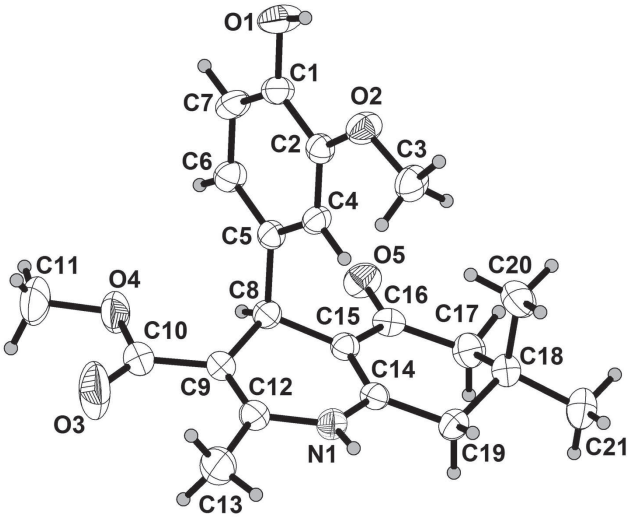
Crystal structure of 4-(4-hydroxy-3-methoxy-phenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydro-quinoline-3-carboxylic acid methyl ester, $C_{21}H_{25}NO_5$



DOI 10.1515/ncrs-2016-0291
Received October 2, 2016; accepted March 16, 2017; available online April 1, 2017

Abstract

$C_{21}H_{25}NO_5$, monoclinic, $P2_1/n$ (no. 14) $a = 10.4501(19)$ Å, $b = 15.750(3)$ Å, $c = 12.043(2)$ Å, $\beta = 102.625(3)°$, $V = 1934.3(6)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0406$, $wR_{ref}(F^2) = 0.1177$, $T = 296(2)$ K.

CCDC no.: 1538499

The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	$0.39 \times 0.35 \times 0.27$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	8.70.9 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	50°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9762, 3406, 0.019
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2764
$N(param)_{refined}$:	249
Programs:	Bruker programs [6], SHELX [7]

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

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.60107(15)	0.17023(10)	0.86364(14)	0.0370(4)
C2	0.71576(15)	0.20068(9)	0.93346(12)	0.0328(3)
C3	0.81016(17)	0.27566(12)	1.10414(15)	0.0463(4)
H3A	0.7848	0.3004	1.1690	0.069*
H3B	0.8747	0.2324	1.1291	0.069*
H3C	0.8464	0.3188	1.0638	0.069*
C4	0.83370(14)	0.19122(9)	0.90157(13)	0.0328(3)
H4A	0.9102	0.2110	0.9492	0.039*
C5	0.84017(14)	0.15249(9)	0.79909(13)	0.0315(3)
C6	0.72555(15)	0.12342(10)	0.73030(13)	0.0382(4)
H6A	0.7279	0.0977	0.6613	0.046*
C7	0.60700(15)	0.13201(11)	0.76252(14)	0.0420(4)
H7A	0.5306	0.1117	0.7152	0.050*
C8	0.97209(14)	0.14147(9)	0.76621(13)	0.0319(3)
H8A	0.9555	0.1249	0.6859	0.038*
C9	1.05293(15)	0.07186(9)	0.83628(13)	0.0354(4)
C10	1.01773(17)	−0.01669(11)	0.80771(17)	0.0468(4)
C11	0.8835(3)	−0.10788(14)	0.6768(3)	0.0973(10)
H11A	0.8176	−0.1053	0.6075	0.146*
H11B	0.9570	−0.1402	0.6644	0.146*
H11C	0.8478	−0.1346	0.7350	0.146*
C12	1.15156(15)	0.09033(10)	0.92517(14)	0.0370(4)

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Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C13	1.23373(19)	0.03010(12)	1.00703(18)	0.0573(5)
H13A	1.2966	0.0615	1.0617	0.086*
H13B	1.1785	−0.0020	1.0456	0.086*
H13C	1.2788	−0.0079	0.9663	0.086*
C14	1.14633(14)	0.23790(9)	0.87189(12)	0.0309(3)
C15	1.04808(14)	0.22405(9)	0.77986(12)	0.0304(3)
C16	1.01231(14)	0.28970(10)	0.69647(13)	0.0340(4)
C17	1.08227(17)	0.37406(10)	0.71625(14)	0.0422(4)
H17A	1.0223	0.4184	0.6817	0.051*
H17B	1.1551	0.3736	0.6782	0.051*
C18	1.13435(16)	0.39564(10)	0.84131(14)	0.0390(4)
C19	1.21672(15)	0.32064(10)	0.89541(14)	0.0386(4)
H19A	1.2965	0.3184	0.8667	0.046*
H19B	1.2415	0.3293	0.9770	0.046*
C20	1.02024(19)	0.41084(12)	0.89935(17)	0.0518(5)
H20A	0.9693	0.4583	0.8643	0.078*
H20B	0.9659	0.3611	0.8917	0.078*
H20C	1.0538	0.4227	0.9786	0.078*
C21	1.2197(2)	0.47474(11)	0.85252(19)	0.0583(5)
H21A	1.1678	0.5221	0.8184	0.088*
H21B	1.2547	0.4864	0.9316	0.088*
H21C	1.2904	0.4657	0.8147	0.088*
H1	0.492(2)	0.1972(16)	0.962(2)	0.086(8)*
N1	1.18819(12)	0.17428(8)	0.94737(11)	0.0366(3)
H1A	1.2394	0.1866	1.0115	0.044*
O1	0.48243(11)	0.17834(9)	0.89232(12)	0.0548(4)
O2	0.69880(10)	0.23930(8)	1.03121(9)	0.0440(3)
O3	1.06367(19)	−0.07877(9)	0.85780(15)	0.0899(6)
O4	0.92575(14)	−0.02315(8)	0.71204(14)	0.0700(5)
O5	0.92788(11)	0.27803(8)	0.60840(9)	0.0458(3)

Source of material

The title compound was synthesized according to a reported procedure [5]. A mixture of 5,5-dimethyl-cyclohexane-1,3-dione (10 mmol), 3-methoxy-4-hydroxy-benzaldehyde (10 mmol), and 3-amino-2-butenoic 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.

Experimental details

Hydrogen atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2U_{eq}(C, N) and 1.5U_{eq} for methyl groups.

Discussion

Several derivatives of 4-arylpolyhydroquinoline are endowed with different types of biological activities [1]. It has been reported that some 4-arylpolyhydroquinoline derivatives exhibit antimicrobial activity, growth stimulating effects, antifungal and plant growth regulation effects, antitumor activity, central nervous system (CNS) activity and hypotensive effect [2, 3]. Moreover, some 4-arylpolyhydroquinoline derivatives are well known for antihistaminic activity, platelet anti-aggregating activity and local anaesthetic activity, antiallergenic effect, antidepressant effect and as antiproliferation agents [4].

In the crystal structure of the title compound, the six-membered ring containing nitrogen atom shows a folding angle of 21.5° between the planes intersecting C9-C12-N1-C14-C15 and C12 -N1C14. The folding of the adjacent hexenone moiety shows a folding angle of 47° between the enone moiety (C17-C16-C15-C14-C19) and the aliphatic part (C19-C18-C17) of the ring (*cf.* see the figure). All geometric parameters are in the expected ranges.

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