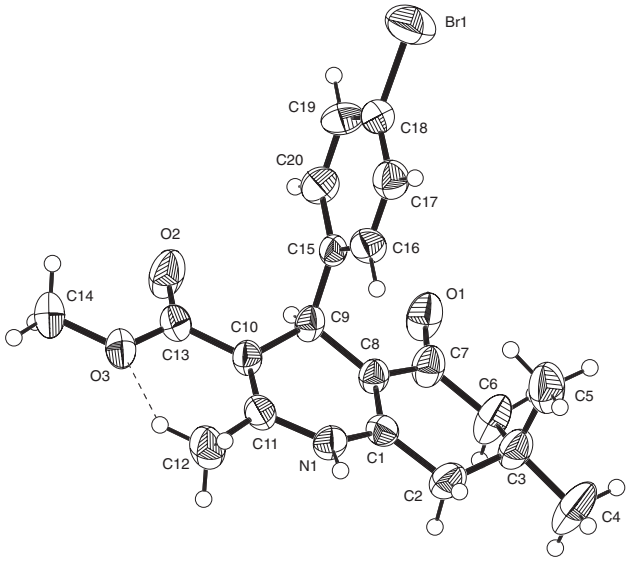
Tao-Wen Ren, Xing-Peng Xiao, Bai-Yu Li, Yu-Mei Zhao and Shao-Peng Peng*

Crystal structure of methyl 4-(4-bromophenyl)-2, 7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate, $C_{20}H_{22}BrNO_3$



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Abstract

$C_{20}H_{22}BrNO_3$, monoclinic, $P2_1/n$ (no. 14), $a = 9.597(2)$ Å, $b = 18.236(5)$ Å, $c = 11.640(3)$ Å, $\beta = 111.355(5)°$, $V = 1897.3(8)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0497$, $wR_{ref}(F^2) = 0.1240$, $T = 296(2)$ K.

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

***Corresponding author: Shao-Peng Peng,** Department of Neurosurgery, Gansu Provincial Hospital, Lanzhou, Gansu, P.R. China, e-mail: shaopeng_peng666@126.com
Tao-Wen Ren, Bai-Yu Li and Yu-Mei Zhao: Cadre Ward 5 of Neurology Department, Gansu Provincial Hospital, Lanzhou, Gansu, P.R. China
Xing-Peng Xiao: Department of Anaesthesiology, Renmin Hospital of Wuhan University, Wuhan, Hubei, P.R. China

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.26 × 0.21 × 0.17 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	2.18 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9434, 3337, 0.056
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1913
$N(param)_{refined}$:	226
Programs:	Olex2 [1], SHELX [2], Bruker [3]

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

Atom	x	y	z	U_{iso}*/U_{eq}
N1	0.2178(3)	0.31642(16)	0.4445(3)	0.0437(8)
H1	0.156682	0.326072	0.371137	0.052*
Br1	−0.04573(7)	0.05860(3)	0.85548(5)	0.0885(3)
O1	0.3753(4)	0.36240(15)	0.8710(3)	0.0718(9)
O2	0.2494(3)	0.45084(13)	0.7455(2)	0.0586(8)
O3	0.5493(3)	0.14298(14)	0.6953(2)	0.0617(8)
C1	0.2667(6)	0.4951(2)	0.8529(4)	0.0731(14)
H1A	0.219089	0.541709	0.827288	0.110*
H1B	0.221228	0.470487	0.903332	0.110*
H1C	0.371173	0.502391	0.899529	0.110*
C2	0.3101(4)	0.3845(2)	0.7672(4)	0.0434(9)
C3	0.2908(4)	0.34053(18)	0.6568(3)	0.0373(9)
C4	0.2202(4)	0.36267(19)	0.5398(3)	0.0407(9)
C5	0.1398(5)	0.43307(19)	0.4947(4)	0.0587(12)
H5A	0.100553	0.433372	0.406107	0.088*
H5B	0.059153	0.437888	0.524358	0.088*
H5C	0.208062	0.473292	0.524477	0.088*
C6	0.3070(4)	0.25692(19)	0.4613(3)	0.0381(9)
C7	0.3819(4)	0.23108(18)	0.5767(3)	0.0366(9)
C8	0.3544(4)	0.26328(18)	0.6859(3)	0.0364(9)
H8	0.451202	0.266870	0.754087	0.044*
C9	0.3177(4)	0.2239(2)	0.3471(3)	0.0484(10)
H9A	0.387764	0.252257	0.322641	0.058*
H9B	0.220577	0.226978	0.280985	0.058*
C10	0.3677(5)	0.1446(2)	0.3633(3)	0.0517(11)
C11	0.4040(6)	0.1193(3)	0.2525(4)	0.0877(17)
H11A	0.316459	0.123751	0.178972	0.132*
H11B	0.482649	0.149193	0.245146	0.132*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H11C	0.435773	0.069010	0.263679	0.132*
C12	0.2409(5)	0.0966(2)	0.3705(4)	0.0712(13)
H12A	0.155698	0.100987	0.295150	0.107*
H12B	0.273115	0.046360	0.382222	0.107*
H12C	0.214154	0.112141	0.438468	0.107*
C13	0.5030(5)	0.1383(2)	0.4811(3)	0.0644(13)
H13A	0.529620	0.086976	0.496357	0.077*
H13B	0.586354	0.163292	0.469829	0.077*
C14	0.4811(4)	0.1699(2)	0.5933(4)	0.0475(10)
C15	0.2546(4)	0.21449(18)	0.7278(3)	0.0359(9)
C16	0.1152(4)	0.1928(2)	0.6472(3)	0.0445(10)
H16	0.082118	0.209436	0.566156	0.053*
C17	0.0239(5)	0.1476(2)	0.6831(4)	0.0502(10)
H17	−0.069257	0.133539	0.627118	0.060*
C18	0.0726(5)	0.1236(2)	0.8025(4)	0.0512(11)
C19	0.2088(6)	0.1441(2)	0.8854(4)	0.0642(13)
H19	0.241086	0.127451	0.966455	0.077*
C20	0.2981(5)	0.1901(2)	0.8473(3)	0.0539(11)
H20	0.390103	0.204856	0.904147	0.065*

Source of material

The title compound was synthesized according to a reported procedure [4]. A mixture of 5,5-dimethyl-cyclohexane-1,3-dione (10 mmol), 4-bromobenzaldehyde (10 mmol), 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 precipitates were sequentially washed with ice-cooled water and ethanol and dried under vacuum.

Experimental details

H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å/0.96 Å and N—H = 0.86 Å with U_{iso}(H) = 1.2 times U_{eq}(C) and 1.2 times U_{eq}(N).

Comment

Neuro-oncology is the study of brain and spinal cord neoplasms, many of which are (at least eventually) very dangerous and life-threatening [5]. Among the malignant brain cancers, gliomas of the brainstem and pons, glioblastoma multiforme, and high-grade (highly anaplastic) astrocytoma are among the worst [6]. 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 [7]. Others are known for anti-histaminic activity, platelet anti-aggregating activity and local anaesthetic activity, antiallergenic effect, antidepressant effect and as anti-glioma agents [8].

In the crystal structure of the title compound (Figure), the six-membered ring containing nitrogen atom is nearly planar and the adjacent ring with the keto group adopts a flattened chair conformation. The bond distances and the bond angles in the title compound are comparable with those of known complexes [9–11], which show a slightly modified substituation scheme. The Br atom in the title compound was replaced by one -NO_2 group, one -OCH_3 group and one Cl atom in the complexes reported in reference [9–11], respectively.

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