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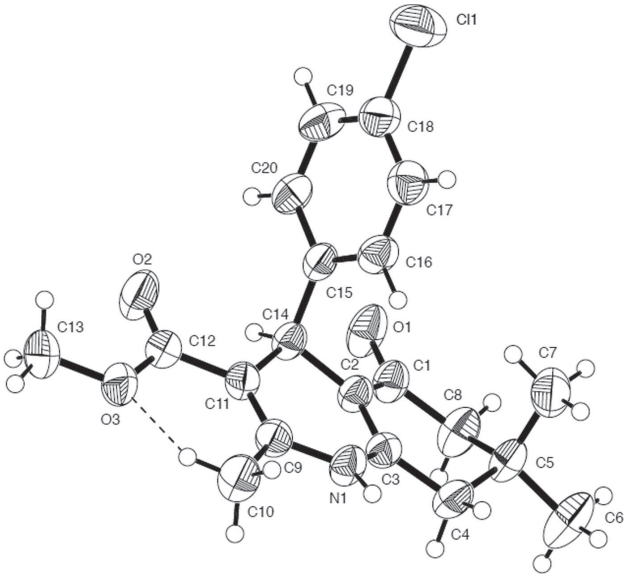
Redetermination of methyl 4-(4-chlorophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate, $C_{20}H_{22}ClNO_3$



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Abstract

$C_{20}H_{22}ClNO_3$, monoclinic, $P2_1/n$ (no. 14), $a = 9.275(5)$ Å, $b = 18.213(9)$ Å, $c = 11.403(6)$ Å, $\beta = 109.718(9)°$, $V = 1813.4(16)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0483$, $wR_{ref}(F^2) = 0.1346$, $T = 296(2)$ K.

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

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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:	0.23 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9051, 3193, 0.041
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2073
$N(param)_{refined}$:	226
Programs:	Olex2 [7], SHELX [8], Bruker [9]

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

Atom	x	y	z	U_{iso}*/U_{eq}
N1	0.7933(2)	0.31280(9)	1.05911(17)	0.0473(5)
H1	0.855568	0.322165	1.132677	0.057*
Cl1	1.06528(11)	0.06073(4)	0.64976(9)	0.0930(3)
O1	0.7478(2)	0.45105(8)	0.75744(16)	0.0601(5)
O2	0.6453(2)	0.35914(9)	0.63209(17)	0.0686(6)
O3	0.4557(2)	0.14229(8)	0.80716(15)	0.0629(5)
C1	0.7304(4)	0.49596(13)	0.6509(3)	0.0725(9)
H1A	0.768419	0.544373	0.677478	0.109*
H1B	0.787173	0.474951	0.602626	0.109*
H1C	0.624040	0.498738	0.600902	0.109*
C2	0.7001(3)	0.38212(12)	0.7357(2)	0.0444(6)
C3	0.7202(3)	0.33737(11)	0.8453(2)	0.0408(5)
C4	0.7898(3)	0.35962(11)	0.9630(2)	0.0437(6)
C5	0.8687(3)	0.43023(12)	1.0083(2)	0.0608(7)
H5A	0.907423	0.430223	1.097695	0.091*
H5B	0.952056	0.436173	0.977120	0.091*
H5C	0.797545	0.469981	0.979284	0.091*
C6	0.7028(3)	0.25290(12)	1.0423(2)	0.0413(5)
C7	0.6289(2)	0.22776(11)	0.9269(2)	0.0398(5)
C8	0.6590(3)	0.26058(11)	0.81633(19)	0.0407(5)
H8	0.560978	0.263460	0.747809	0.049*
C9	0.5264(3)	0.16696(12)	0.9103(2)	0.0458(6)
C10	0.5054(3)	0.13319(14)	1.0223(2)	0.0591(7)
H10A	0.479943	0.081721	1.005349	0.071*
H10B	0.419258	0.156718	1.037150	0.071*
C11	0.6437(3)	0.13899(13)	1.1390(2)	0.0499(6)
C12	0.7731(3)	0.09286(14)	1.1280(2)	0.0680(8)
H12A	0.742082	0.042310	1.117312	0.102*
H12B	0.799083	0.108854	1.057463	0.102*
H12C	0.860601	0.097988	1.202333	0.102*
C13	0.6047(4)	0.11307(17)	1.2518(2)	0.0818(9)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H13A	0.522141	0.142132	1.259836	0.123*
H13B	0.574408	0.062417	1.241197	0.123*
H13C	0.692912	0.118335	1.325583	0.123*
C14	0.6896(3)	0.21877(12)	1.1559(2)	0.0506(6)
H14A	0.614404	0.245676	1.180781	0.061*
H14B	0.787300	0.222909	1.222723	0.061*
C15	0.7650(3)	0.21232(11)	0.77410(19)	0.0404(5)
C16	0.9043(3)	0.19047(12)	0.8539(2)	0.0486(6)
H16	0.936072	0.206917	0.935874	0.058*
C17	0.9992(3)	0.14495(12)	0.8170(2)	0.0552(7)
H17	1.094280	0.131349	0.872809	0.066*
C18	0.9519(3)	0.12029(13)	0.6982(3)	0.0566(7)
C19	0.8149(4)	0.14146(14)	0.6166(3)	0.0695(8)
H19	0.783437	0.124604	0.534863	0.083*
C20	0.7227(3)	0.18762(13)	0.6543(2)	0.0575(7)
H20	0.629407	0.202428	0.597188	0.069*

Source of material

The title compound was synthesized according to a reported procedure [6]. A mixture of 5,5-dimethyl-cyclohexane-1,3-dione (10 mmol), 4-chlorobenzaldehyde (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 the precipitates, they were sequentially washed with ice-cooled water and ethanol and then dried under a vacuum.

Experimental details

H atoms bonded to C and N atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 or 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

Several derivatives of 4-arylpolyhydroquinoline are endowed with different types of biological activities [1]. It has been reported that 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 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 (*cf*. the Figure), the six-membered ring containing nitrogen atom is nearly planar with the C14 atom folded out of the mean plane. The neighboring six-membered ring adopts a flattened chair conformation. The quinoline moiety is almost perpendicular to the chlorophenyl group. The crystal structure of the title compound is the same as the previously reported compounds 3-carboxymethyl-4-(4-chlorophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoleine [5]. Although the two structure are the same, the crystal quality and crystal parameters of the title compound is better than those in previously published one (for the literature known structure only 2791 reflections were measured and 1004 of them are observed ($I > 2s(I)$). Most importantly, the methods of their synthsis are very different.

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