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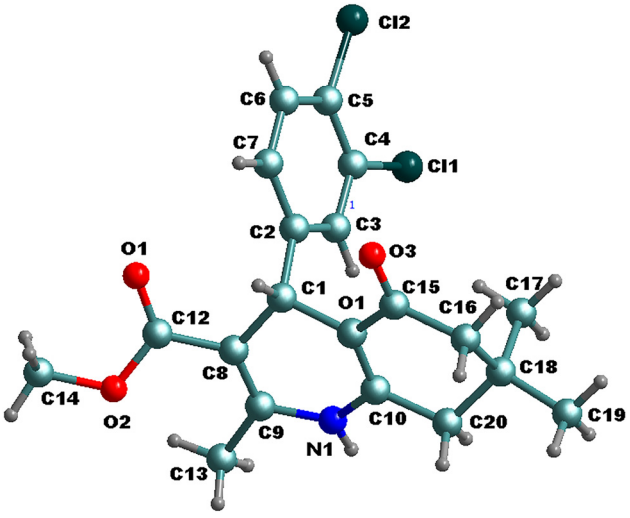
The crystal structure of methyl 4-(3,4-dichlorophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate, $C_{20}H_{21}Cl_2NO_3$



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Abstract

$C_{20}H_{21}Cl_2NO_3$, orthorhombic, *Pbca* (no. 61), a = 14.938(3) Å, b = 13.224(3) Å, c = 18.753(4) Å, V = 3704.3(12) Å^3, Z = 8, R_{gt} (F) = 0.0462, wR_{ref} (F^2) = 0.1435, T = 296(2) K.

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The molecular structure is shown in the figure. Table 1 contains the crystallographic data and the list of the atoms including atomic coordinates and displacement parameters can be found in the cif-file attached to this article.

1 Source of materials

All starting materials were commercial and used without any further purification. The title compound was synthesized as follows with reference 5: A mixture of 20 mmol 5,5-dimethyl-1,3-cyclohexanedione, 20 mmol 3,4-dichlorobenzaldehyde, 20 mmol methyl acetoacetate, 30 mmol ammonium acetate in 20 mL methanol was stirred for 5 min, then 2 mmol *p*-toluenesulfonic acid used as catalyst was added. The mixture was stirred for 6 h at room temperature, then washed with saturated sodium bicarbonate, and filtered. The single crystal was obtained by recrystallizing the crude product from an ethanol solution.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.29 × 0.24 × 0.18 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.37 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω scans
θ_{max}, completeness:	28.7°, 100 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	22656, 4718, 0.076
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3,175
$N(param)_{refined}$:	239
Programs:	Bruker,[1] Olex2,[2] SHELX[3,4]

2 Experimental details

The structure was solved by Direct Methods with the SHELXS-2018 program. All H-atoms from C and other atoms were positioned with idealized geometry and refined isotropically (U_{iso} (H) = 1.2 U_{eq} (C) or U_{eq} (N)) using a riding model with C–H = 0.930, 0.960, 0.970 and 0.980 Å, and N–H = 0.860 Å.

3 Comment

Due to the similar activity of polyhydroquinoline derivatives to 1,4-dihydropyridine derivatives and their pharmacological activities such as calcium ion regulation, the synthesis of hexahydroquinoline derivatives has become an important aspect of research on 1,4-dihydropyridine derivatives. To date, many crystal structures of methyl 2,7,7-trimethyl-5-oxo-

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4-phenyl-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate[5] and its derivatives of chloride-substituted on 4-phenyl group have been reported elsewhere, such as methyl 4-(2,4-dichlorophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate,[6] methyl 4-(2-chloro-5-nitrophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate,[7] methyl 2,7,7-trimethyl-5-oxo-4-(3-chlorophenyl)-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate,[8,9] 3-(methoxycarbonyl)-4-(4-chlorophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline.[10,11]

The title compound crystallizes in an orthorhombic space group, *Pbca* (no. 61), with the fomula of $C_{20}H_{21}Cl_2NO_3$. The structure consists of one hexahydroquinoline ring (including one dihydropyridine ring and one cyclohexenone ring) and one dichloro-substituted benzene ring. The dihydropyridine ring adapts a flattened boat configuration. The cyclohexenone ring has a sofa-like conformation. The benzene ring is approximately planar. There is one 1D chain generated by a type of intermolecular hydrogen bond, N1–H1···O3. All bond lengths of the title compound are similar to the reported results.[5–11]

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