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Crystal structure of ethyl 4-(3-chlorophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate, $C_{21}H_{24}ClNO_3$

Table 1: Data collection and handling.

Crystal:	Block, yellow
Size:	0.32 × 0.28 × 0.25 mm
Wavelength:	Mo K α radiation ($\lambda = 0.71073$ Å)
μ :	0.220 mm ⁻¹
Diffractometer, scan mode:	CCD area detector, Φ and ω -scans
$2\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	18525, 3380, 0.0541
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 2176
$N(\text{param})_{\text{refined}}$:	237
Programs:	Bruker programs [1], OLEX2 [2], SHELX [3]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.38417(14)	0.07907(15)	0.67038(16)	0.0480(6)
H1	0.382670	0.069075	0.730163	0.058
Cl1	0.07594(6)	0.03553(7)	0.64578(8)	0.0899(4)
O1	0.34465(15)	0.26977(15)	0.42352(19)	0.0750(8)
O2	0.39095(18)	0.32592(16)	0.5537(2)	0.0915(9)
O3	0.38800(14)	-0.03147(13)	0.36612(13)	0.0575(6)
C1	0.1287(2)	0.0655(2)	0.5469(3)	0.0605(9)
C2	0.0902(2)	0.0811(2)	0.4651(3)	0.0760(11)
H2	0.036355	0.076576	0.462149	0.091
C3	0.1322(3)	0.1035(3)	0.3872(3)	0.0866(13)
H3	0.106708	0.113216	0.330203	0.104
C4	0.2129(2)	0.1120(2)	0.3916(3)	0.0724(11)
H4	0.240465	0.127766	0.337859	0.087
C5	0.25177(18)	0.09724(17)	0.4751(2)	0.0484(7)
C6	0.20789(17)	0.07292(19)	0.5530(2)	0.0529(8)
H6	0.232581	0.061488	0.610167	0.063
C7	0.33959(17)	0.10569(18)	0.4830(2)	0.0446(7)
H7	0.360218	0.118024	0.419830	0.053
C8	0.36302(17)	0.17781(18)	0.5485(2)	0.0451(7)
C9	0.36804(19)	0.2649(2)	0.5126(3)	0.0558(8)
C10	0.3490(3)	0.3533(3)	0.3802(4)	0.1065(17)
H10A	0.348447	0.396490	0.429080	0.128
H10B	0.397441	0.358476	0.345519	0.128
C11	0.2884(3)	0.3660(3)	0.3203(4)	0.1195(18)
H11A	0.292420	0.421430	0.292463	0.179
H11B	0.289423	0.323771	0.271296	0.179
H11C	0.240503	0.361728	0.354732	0.179
C12	0.37905(17)	0.16208(18)	0.6394(2)	0.0467(7)
C13	0.3931(2)	0.2265(2)	0.7154(3)	0.0688(10)
H13A	0.397010	0.198357	0.775466	0.103

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Abstract

$C_{21}H_{24}ClNO_3$, orthorhombic, *Pbcn* (no. 60), $a = 17.215(11)$ Å, $b = 15.749(11)$ Å, $c = 14.136(9)$ Å, $V = 3832(4)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.058$, $wR_{\text{ref}}(F^2) = 0.177$, $T = 296(2)$ K.

CCDC no.: 1847962

Source of materials

The title compound was synthesized according to reported procedures. A mixture of 1,1-dimethyl-3,5-cyclohexanedione (10 mmol), 3-chlorobenzaldehyde (10 mmol), ammonium acetate (10 mmol) and ethyl acetoacetate (10 mmol) in ethanol (100 mL) was refluxed for 2–3 h and then cooled to room temperature. After filtering the precipitates, they were

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H13B	0.350662	0.266055	0.716945	0.103
H13C	0.440521	0.256363	0.702563	0.103
C14	0.39150(15)	0.01270(17)	0.60964(18)	0.0403(7)
C15	0.37588(16)	0.02432(17)	0.51714(18)	0.0391(6)
C16	0.39399(16)	−0.04244(18)	0.45158(19)	0.0412(7)
C17	0.42347(18)	−0.12548(18)	0.4897(2)	0.0487(7)
H17A	0.479701	−0.125556	0.485240	0.058
H17B	0.404362	−0.170761	0.449382	0.058
C18	0.40105(17)	−0.14539(18)	0.5907(2)	0.0459(7)
C19	0.42054(17)	−0.06797(18)	0.65033(19)	0.0452(7)
H19A	0.398411	−0.075298	0.712900	0.054
H19B	0.476498	−0.064294	0.657312	0.054
C20	0.3145(2)	−0.1664(2)	0.5974(3)	0.0685(10)
H20A	0.301358	−0.178886	0.661964	0.103
H20B	0.284591	−0.118732	0.575994	0.103
H20C	0.303248	−0.214905	0.558603	0.103
C21	0.4485(2)	−0.2213(2)	0.6259(3)	0.0715(10)
H21A	0.434279	−0.234022	0.690008	0.107
H21B	0.438095	−0.269768	0.586607	0.107
H21C	0.502821	−0.207860	0.623059	0.107

sequentially washed with ice-cooled water and ethanol and then dried under vacuum.

Experimental details

H atoms were positioned geometrically and refined using a riding model, with C—H = 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

4-Arylpolyhydroquinolines possess diverse pharmacological and biological activities such as antitumor, analgesic and ulcerogenic, anti-inflammatory, anticoagulant, phototriggering, and fungicidal properties, and can act as anticoagulants in the production of pesticides [4]. Neurofibromatosis type I (NF-1) is a complex multi-system human disorder caused by the mutation of ageneonchromosome 17 that is responsible for production of a protein called neurofibromin which is needed for normal function in many human cell types [5]. NF-1 causes tumors along the nervous system which can grow anywhere on the body. NF-1 is one of the most common genetic disorders and is not limited to any person's race or sex [6]. Recent research has also indicated that these heterocycles is an effective treatment to shrink NF-1 and control symptoms [7].

In the crystal structure of the title compound (Figure), the six-membered ring containing nitrogen atom is slightly folded and the adjacent ring containing the keto group adopts a flattened chair conformation. The nitrogenous heterocyclic is almost perpendicular to the chlorophenyl moiety and is almost coplanar with the mean plane of the ketone ring. The bond distances and the bond angles in the title compound are comparable with those in known compounds [8–10]. Compared with the known similar structures [8–10], the main difference is that they have different functional groups. The Cl atom in the title compound was replaced by one Br atom, one -OCH₃ group and one H atom in the compounds reported in reference 5, 6 and 7, respectively.

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