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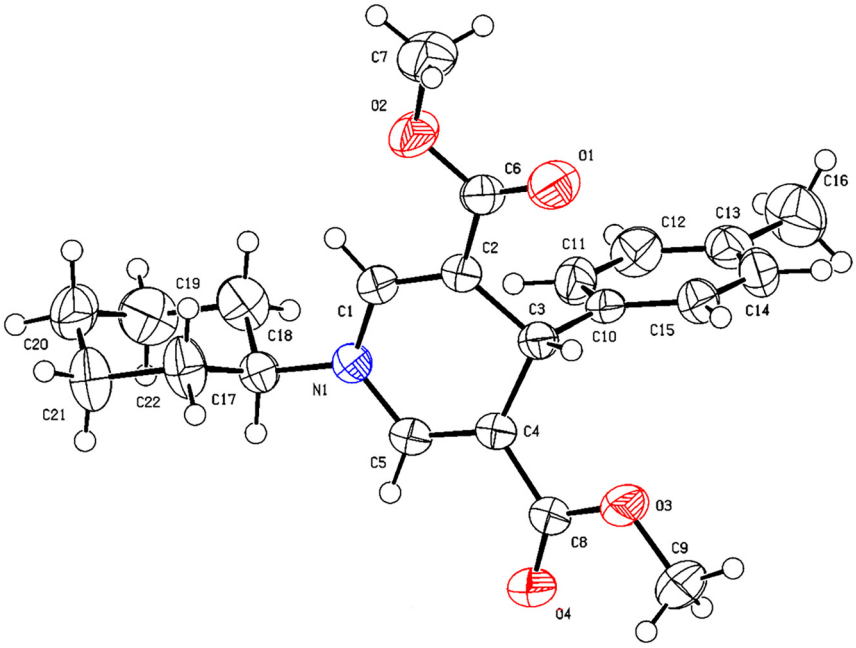
Crystal structure of 1-cyclohexyl-4-*p*-tolyl-1,4-dihydropyridine-3,5-dicarboxylic acid dimethyl ester, $C_{22}H_{27}NO_4$



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Abstract

$C_{22}H_{27}NO_4$, triclinic, $P\bar{1}$ (no. 2), a = 9.3356(8) Å, b = 10.2842(11) Å, c = 11.5902(12) Å, α = 103.266(4)°, β = 106.189(3)°, γ = 97.779(4)°, V = 1016.20(18) Å^3, Z = 2, $R_{gt}(F)$ = 0.0413, w$R_{ref}(F^2)$ = 0.0928, T = 293 K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.25 × 0.23 × 0.20 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.08 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}:	27,085, 3,566, 0.044
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3,180
$N(param)_{refined}$:	247
Programs:	Bruker,[1,2] SHELX[3,4]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.94039 (16)	0.25010 (14)	0.45807 (13)	0.0405 (3)
H1	1.045627	0.278560	0.495346	0.049*
C2	0.87877 (15)	0.25380 (13)	0.33990 (12)	0.0366 (3)
C3	0.70787 (15)	0.22522 (14)	0.27723 (12)	0.0367 (3)
H3	0.683159	0.168153	0.191095	0.044*
C4	0.63172 (15)	0.14576 (14)	0.34767 (13)	0.0382 (3)
C5	0.70392 (15)	0.14563 (14)	0.46500 (13)	0.0404 (3)
H5	0.649160	0.102565	0.506657	0.048*
C6	0.97439 (16)	0.28364 (14)	0.26437 (13)	0.0409 (3)
C7	1.2264 (2)	0.3446 (2)	0.26052 (18)	0.0707 (5)
H7A	1.220462	0.261508	0.199480	0.106*
H7B	1.328872	0.376870	0.317312	0.106*
H7C	1.198218	0.412492	0.219013	0.106*
C8	0.47245 (16)	0.07021 (15)	0.28922 (14)	0.0437 (3)
C9	0.2743 (2)	−0.0260 (2)	0.09521 (19)	0.0812 (7)
H9A	0.266644	−0.117248	0.103395	0.122*
H9B	0.252897	−0.029066	0.008553	0.122*
H9C	0.201937	0.015851	0.127018	0.122*
C10	0.64964 (14)	0.35639 (14)	0.27381 (12)	0.0364 (3)
C11	0.67890 (18)	0.46035 (16)	0.38298 (14)	0.0484 (4)
H11	0.736839	0.450582	0.458786	0.058*
C12	0.62356 (19)	0.57810 (17)	0.38112 (16)	0.0557 (4)
H12	0.645306	0.646385	0.455590	0.067*
C13	0.53646 (18)	0.59605 (17)	0.27045 (17)	0.0528 (4)
C14	0.50708 (18)	0.49276 (18)	0.16245 (16)	0.0549 (4)
H14	0.448130	0.502279	0.086873	0.066*
C15	0.56313 (17)	0.37478 (16)	0.16342 (14)	0.0465 (4)
H15	0.542117	0.307146	0.088654	0.056*
C16	0.4758 (3)	0.7247 (2)	0.2686 (2)	0.0862 (7)
H16A	0.519028	0.772240	0.219800	0.129*
H16B	0.503191	0.782540	0.352576	0.129*
H16C	0.366667	0.701122	0.232092	0.129*
C17	0.92787 (16)	0.20593 (15)	0.65749 (13)	0.0427 (3)
H17	0.846732	0.168943	0.686919	0.051*
C18	1.0000 (2)	0.34911 (17)	0.74224 (15)	0.0599 (4)
H18A	0.923310	0.404367	0.737546	0.072*
H18B	1.079021	0.390275	0.714243	0.072*
C19	1.0694 (3)	0.3471 (2)	0.87780 (16)	0.0717 (5)
H19A	1.120664	0.439092	0.929443	0.086*
H19B	0.988584	0.315542	0.908776	0.086*
C20	1.1817 (2)	0.2547 (2)	0.88788 (16)	0.0689 (5)
H20A	1.268611	0.292667	0.866703	0.083*
H20B	1.218284	0.250514	0.973645	0.083*
C21	1.1115 (2)	0.1131 (2)	0.80287 (16)	0.0680 (5)
H21A	1.033212	0.070668	0.830861	0.082*
H21B	1.189118	0.058852	0.807317	0.082*
C22	1.0413 (2)	0.11418 (18)	0.66790 (15)	0.0610 (5)
H22A	1.121438	0.145755	0.636349	0.073*
H22B	0.990345	0.021862	0.616790	0.073*
N1	0.85504 (13)	0.20614 (13)	0.52659 (11)	0.0436 (3)
O1	0.92580 (13)	0.27710 (14)	0.15519 (10)	0.0639 (3)
O2	1.12405 (12)	0.31900 (13)	0.32894 (10)	0.0590 (3)
O3	0.42612 (13)	0.05273 (12)	0.16516 (10)	0.0612 (3)
O4	0.39079 (12)	0.02697 (13)	0.34210 (11)	0.0618 (3)

1 Source of material

4–Methylbenzaldehyde (6.5043 g, 0.0541 mol), methyl propiolate (9.1022 g, 0.1082 mol) and acetic acid (catalytic dose) were added to a 500 ml three-necked flask. After the reaction solution was heated to 90 °C, cyclohexylamine (5.3685 g, 0.0541 mol) was added and refluxed for 3 h, then naturally cooled to room temperature. A yellow precipitate was collected and recrystallized from 95 % ethanol, yield 73.5 %. Yellow block crystals were obtained from the filtrate by recrystallization.

2 Experimental details

Using Bruker APEX2 and Bruker SAINT,[1] the structure was solved with the Sheletl (Bruker, 2008)[2] structure solution program and refined with the Shelxl2018/3 (Sheldrick, 2018)[3,4] refinement package.

3 Comment

1,4–Dihydropyridine is a very important class of nitrogen-containing heterocyclic compounds, which has been widely used in biology, medicine and organic catalysis.[5] So far, many attempts have been made by researchers to synthesize them, such as ultrasonic method,[6] metal catalysis,[7] nano catalysis,[8] acid catalysis,[9] enzyme catalysis[10] and so on.

The title compound crystallizes in the triclinic space group, $P\bar{1}$. The conformational details and atom numbering of the title compound are shown in the Figure 1. 1,4–Dihydropyridine ring adopts a flat-boat conformation when the two methoxycarbonyl groups are twisted in opposite directions. The plane of the 4-p-tolylgroup in this compound is approximately perpendicular to the 1,4-dihydropyridine ring. In this crystal the molecules of the title compound form centrosymmetric dimers due to the C12–H12···O2 hydrogen bond (H···C = 2.718 Å, C–H···O = 126.29°). These dimers furthermore generate infinite chains via weak C(17)–H(17)···O4 (H···O = 2.675 Å, C–H···O = 150.39°) and C5–H5···O4 (H···O = 2.508 Å, C–H···O = 156.46°) intermolecular hydrogen bonds. The bond lengths and bond angles of the crystal structure are within a reasonable range.[11–17]

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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