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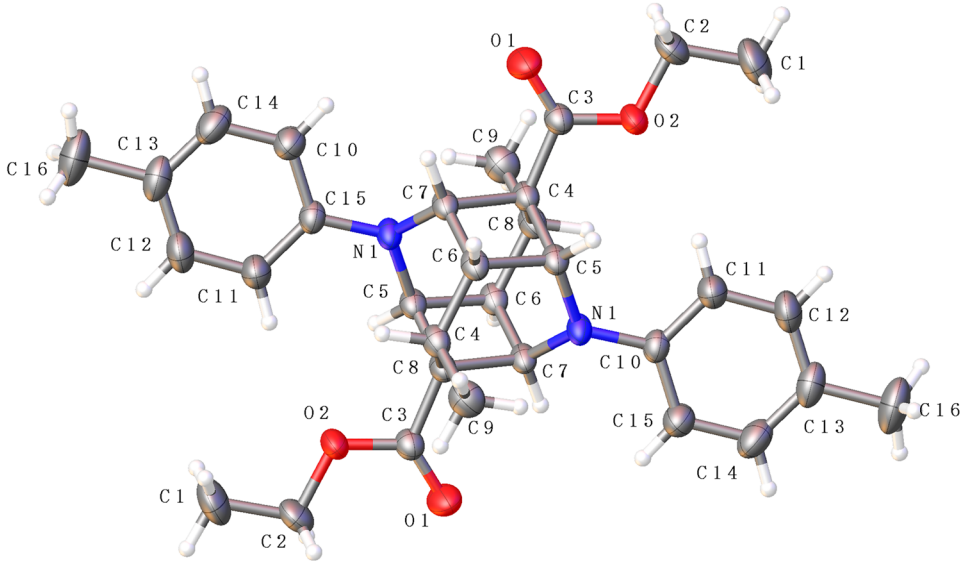
Crystal structure of diethyl 6,12-dimethyl-3,9-di-p-tolyl-3,9-diazapentacyclo[6.4.0.0^{2,7}.0^{4,11}.0^{5,10}] dodecane-1,5-dicarboxylate, $C_{32}H_{38}N_2O_4$



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Abstract

$C_{15}H_{14}N_2O$, triclinic, $P\bar{1}$ (no. 2), a = 6.983(5) Å, b = 8.472(6) Å, c = 12.113(7) Å, β = 73.45°, V = 679.29(8) Å^3, Z = 1, $R_{gt}(F)$ = 0.0566, $wR_{ref}(F^2)$ = 0.1875, T = 296 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.

Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.18 × 0.16 × 0.15 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.08 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.6°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4252, 3054, 0.026
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 2416
$N(param)_{refined}$:	175
Programs:	Bruker [1], SHELX [2, 3]

Source of material

Dissolve 1,4-diphenyl-1,4-dihydropyridine-3-carboxylic acid ethyl ester (10 mmol) in 50 mL of methanol: tetrahydrofuran = 1:1 mixed solution, and add to a 100 mL

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.02312 (17)	0.28526 (16)	0.46273 (11)	0.0514 (4)
O2	0.29314 (16)	0.35214 (14)	0.33028 (9)	0.0426 (3)
N1	0.30503 (18)	0.00094 (15)	0.64789 (10)	0.0319 (3)
C1	0.2940 (4)	0.5395 (3)	0.18096 (18)	0.0720 (7)
H1A	0.213974	0.622278	0.151057	0.108*
H1B	0.354800	0.458629	0.123087	0.108*
H1C	0.397681	0.583680	0.202300	0.108*
C2	0.1630 (3)	0.4678 (2)	0.28415 (15)	0.0496 (5)
H2A	0.093260	0.549674	0.341189	0.060*
H2B	0.063264	0.416255	0.262729	0.060*
C3	0.2016 (2)	0.26758 (18)	0.42067 (12)	0.0331 (4)
C4	0.3522 (2)	0.15220 (17)	0.46035 (12)	0.0301 (3)
C5	0.4799 (2)	0.02059 (17)	0.36868 (12)	0.0301 (3)
H5	0.445168	0.032310	0.295651	0.036*
C6	0.3727 (2)	−0.10906 (17)	0.44699 (11)	0.0308 (3)
H6	0.282916	−0.155574	0.412076	0.037*
C7	0.2545 (2)	0.01883 (17)	0.53912 (11)	0.0299 (3)
H7	0.108634	0.029251	0.550152	0.036*
C8	0.4892 (2)	0.23705 (17)	0.51030 (12)	0.0324 (4)
H8	0.574127	0.293154	0.447073	0.039*
C9	0.3729 (3)	0.3600 (2)	0.60231 (15)	0.0441 (4)
H9A	0.465995	0.411599	0.628355	0.066*
H9B	0.289547	0.307984	0.666011	0.066*
H9C	0.289531	0.437944	0.570672	0.066*
C10	0.1737 (2)	−0.06464 (17)	0.74303 (11)	0.0317 (3)
C11	0.2414 (3)	−0.1815 (2)	0.81168 (14)	0.0447 (4)
H11	0.377096	−0.224097	0.792268	0.054*
C12	0.1075 (3)	−0.2351 (2)	0.90925 (15)	0.0525 (5)
H12	0.157013	−0.312215	0.954479	0.063*
C13	−0.0947 (3)	−0.1789 (2)	0.94147 (14)	0.0496 (5)
C14	−0.1624 (3)	−0.0653 (3)	0.87170 (15)	0.0527 (5)
H14	−0.299115	−0.026280	0.890073	0.063*
C15	−0.0313 (2)	−0.0079 (2)	0.77470 (14)	0.0438 (4)
H15	−0.081587	0.069837	0.730201	0.053*
C16	−0.2368 (4)	−0.2359 (3)	1.05037 (17)	0.0748 (7)
H16A	−0.372670	−0.215889	1.045017	0.112*
H16B	−0.227379	−0.179727	1.115682	0.112*
H16C	−0.200175	−0.348247	1.059279	0.112*

Quartz flask. Place the reaction solution in an LED photoreactor with a wavelength of 365 nm, put it in a freezer, and irradiate it under the protection of nitrogen. After reacting for one week, the product precipitates out and is filtered to obtain the title compound.

Experimental details

All hydrogen atoms were placed in the calculated positions and all the non-hydrogen atoms were refined anisotropically.

Comment

The [2+2] -photocyclization reaction is an important type of organic photocyclization reaction. Under the induction of visible light or ultraviolet light, an addition reaction occurs between the unsaturated double bonds of two molecules, turning the double bonds into single bonds. There are two unsaturated double bonds in the molecular structure of 1,4-dihydropyridine compounds. Under light induction [2+2] photocyclization reaction can occur to generate 3,9-diazatetrastarane with inversion symmetry. Some *cis*/*trans* tetrahydrocyclobutane dipyridine compounds show pharmacological activities in the field of anti-HIV and anti-tumor [4, 6–8]. The title structure is shown in the figure. The asymmetric unit is defined as one half of the title molecule. The bond lengths and angles are in the expected ranges [5, 9].

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