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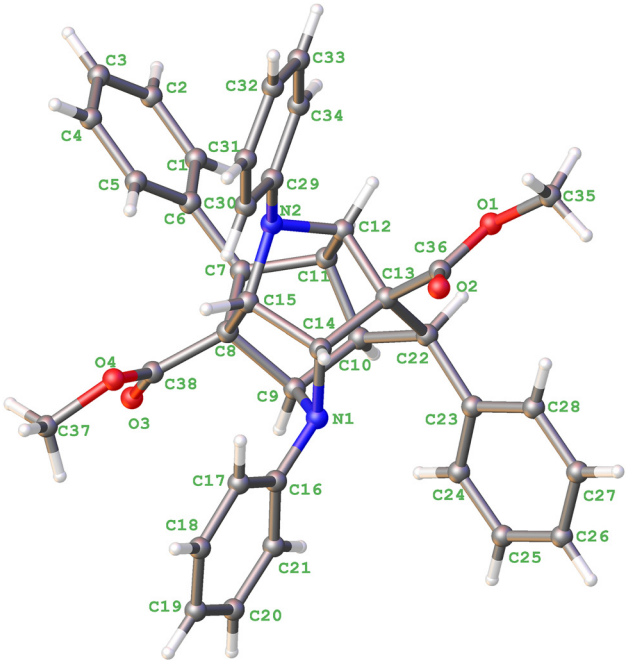
Crystal structure of dimethyl 1,4,6,9-tetraphenylhexahydro-3*H*-2,7,3,5-(epimethanetriyliminomethanetriyl)cyclopenta[*b*]pyridine-3,7(2*H*)-dicarboxylate, $C_{38}H_{34}N_2O_4$



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Abstract

$C_{38}H_{34}N_2O_4$, triclinic, $P\bar{1}$ (no. 2), a = 11.1158(19) Å, b = 11.302(2) Å, c = 12.634(2) Å, α = 72.109(2)°, β = 77.410(2)°, γ = 82.648(3)°, V = 1362.4(5) Å^3, Z = 2, $R_{gt}(F)$ = 0.0473, $wR_{ref}(F^2)$ = 0.1738, T = 296 K.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.18 × 0.16 × 0.15 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.5°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9220, 6596, 0.016
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 5121
$N(\text{param})_{refined}$:	399
Programs:	Bruker [1], SHELX [2, 3]

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

Source of material

Preparation of the 1,4-diaryl-1,4-dihydropyridine-3-carboxylic acid photoreactive raw materials was performed by a literature method [4]: aniline (0.5 mmol), ethyl propiolate (0.5 mmol) and 4-methylcinnamaldehyde (0.5 mmol); piperazine (0.25 mmol) and *p*-toluenesulfonic acid (0.02 mmol) used as catalysts, and 20 mL of 1,2-dichloroethane (20 mL) were mixed. The mixture was heated to reflux for 12 h to obtain 1-phenyl-4-(4-methyl-phenyl)-1,4-dihydroethylpyridine-3-carboxylate. The 1-phenyl-4-(4-methyl-phenyl)-1,4-dihydroethylpyridine-3-carboxylate (0.5 mmol) obtained in the first step was placed near a blue LED (365 nm) for a photoreaction for 3 h [5]. The product was obtained by column chromatography eluting with petroleum ether and ethyl acetate 10:1. The resulting solution was evaporated to get some colorless crystals.

Experimental details

All hydrogen atoms were placed in the calculated positions.

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

Atom	x	y	z	U_{iso}*/U_{eq}
O1	0.43060 (10)	0.33694 (12)	0.25888 (11)	0.0486 (3)
O2	0.41268 (10)	0.44641 (12)	0.38234 (10)	0.0492 (3)
O3	1.07900 (12)	0.56210 (16)	0.16452 (12)	0.0692 (5)
O4	0.98680 (11)	0.54287 (13)	0.34253 (10)	0.0536 (4)
N1	0.71403 (11)	0.63020 (11)	0.21005 (10)	0.0307 (3)
N2	0.74671 (10)	0.30828 (11)	0.35398 (10)	0.0302 (3)
C1	1.04233 (16)	0.17146 (17)	0.15810 (18)	0.0514 (5)
H1	1.030837	0.194130	0.083546	0.062*
C2	1.11000 (19)	0.06188 (19)	0.1988 (2)	0.0656 (6)
H2	1.142688	0.011300	0.152270	0.079*
C3	1.12887 (18)	0.0278 (2)	0.3081 (2)	0.0717 (7)
H3	1.174450	−0.045941	0.336023	0.086*
C4	1.07979 (17)	0.1037 (2)	0.3770 (2)	0.0623 (6)
H4	1.092537	0.080719	0.451179	0.075*
C5	1.01157 (15)	0.21393 (18)	0.33553 (16)	0.0491 (4)
H5	0.979509	0.264685	0.382042	0.059*
C6	0.99089 (13)	0.24892 (15)	0.22523 (14)	0.0396 (4)
C7	0.92002 (13)	0.36777 (14)	0.17107 (12)	0.0348 (3)
H7	0.972447	0.405381	0.097995	0.042*
C8	0.88342 (12)	0.47221 (14)	0.22896 (12)	0.0319 (3)
C9	0.81264 (13)	0.57168 (14)	0.14110 (11)	0.0324 (3)
H9	0.867530	0.632637	0.085875	0.039*
C10	0.75604 (13)	0.49305 (15)	0.08349 (12)	0.0345 (3)
H10	0.785948	0.514756	0.001409	0.041*
C11	0.79379 (13)	0.35783 (14)	0.14407 (12)	0.0337 (3)
H11	0.798609	0.302025	0.097085	0.040*
C12	0.69248 (12)	0.32287 (13)	0.25424 (11)	0.0306 (3)
H12	0.650261	0.249372	0.260418	0.037*
C13	0.60202 (12)	0.44331 (13)	0.24500 (11)	0.0299 (3)
C14	0.66390 (12)	0.52218 (13)	0.29893 (11)	0.0284 (3)
H14	0.607939	0.544653	0.362330	0.034*
C15	0.77867 (12)	0.43449 (13)	0.33603 (11)	0.0289 (3)
H15	0.800076	0.445776	0.403415	0.035*
C16	0.74712 (13)	0.73169 (13)	0.23860 (12)	0.0322 (3)
C17	0.73080 (15)	0.73823 (16)	0.34895 (14)	0.0416 (4)
H17	0.696713	0.673274	0.409280	0.050*
C18	0.76563 (18)	0.84230 (18)	0.36893 (16)	0.0548 (5)
H18	0.753605	0.846053	0.443050	0.066*
C19	0.81710 (17)	0.93929 (18)	0.28242 (17)	0.0558 (5)
H19	0.841967	1.007025	0.297458	0.067*
C20	0.83132 (18)	0.93455 (17)	0.17267 (17)	0.0546 (5)
H20	0.865425	1.000005	0.112908	0.065*
C21	0.79526 (16)	0.83324 (16)	0.15077 (14)	0.0454 (4)
H21	0.803199	0.832644	0.076115	0.054*
C22	0.61473 (13)	0.50144 (14)	0.11513 (12)	0.0337 (3)
H22	0.585652	0.441173	0.086356	0.040*
C23	0.54602 (15)	0.62529 (15)	0.07012 (13)	0.0390 (4)
C24	0.60661 (18)	0.72672 (19)	−0.00652 (17)	0.0575 (5)
H24	0.691986	0.719685	−0.029116	0.069*
C25	0.5418 (2)	0.8377 (2)	−0.0495 (2)	0.0786 (7)
H25	0.584379	0.904547	−0.099898	0.094*
C26	0.4155 (2)	0.8509 (2)	−0.0191 (2)	0.0747 (7)
H26	0.372717	0.926176	−0.047961	0.090*
C27	0.3534 (2)	0.7514 (2)	0.05451 (18)	0.0615 (5)
H27	0.267785	0.758896	0.075170	0.074*
C28	0.41778 (16)	0.63963 (18)	0.09822 (14)	0.0470 (4)
H28	0.374321	0.572757	0.147426	0.056*
C29	0.68045 (12)	0.24302 (13)	0.46162 (12)	0.0292 (3)
C30	0.65222 (14)	0.29058 (15)	0.55398 (13)	0.0376 (3)
H30	0.675482	0.369469	0.546073	0.045*
C31	0.58983 (16)	0.22175 (18)	0.65769 (14)	0.0476 (4)
H31	0.572065	0.254901	0.718665	0.057*
C32	0.55378 (16)	0.10502 (18)	0.67175 (15)	0.0500 (4)
H32	0.510939	0.059688	0.741309	0.060*
C33	0.58199 (16)	0.05666 (16)	0.58164 (16)	0.0488 (4)
H33	0.558509	−0.022423	0.590529	0.059*
C34	0.64494 (15)	0.12373 (15)	0.47752 (14)	0.0406 (4)
H34	0.663776	0.088962	0.417553	0.049*
C35	0.30507 (16)	0.3035 (2)	0.30004 (18)	0.0573 (5)
H35A	0.250371	0.373478	0.271723	0.086*
H35B	0.288198	0.279568	0.381335	0.086*
H35C	0.292657	0.234958	0.274590	0.086*
C36	0.47204 (13)	0.41176 (14)	0.30550 (12)	0.0328 (3)
C37	1.0814 (2)	0.6115 (3)	0.3553 (2)	0.0775 (7)
H37A	1.160365	0.566957	0.343338	0.116*
H37B	1.063779	0.620733	0.430278	0.116*
H37C	1.082978	0.692274	0.300708	0.116*
C38	0.99411 (14)	0.52876 (15)	0.24132 (13)	0.0377 (3)

Comment

Cage dimer 4-aryl-1,4-dihydropyridines have a wide range of biological activities, such as anti-HIV and as an anti-multidrug resistance modulator [6–8]. The caged dimeric compound similar to the title compound has also been reported, which is obtained by a dimerization by photoreaction. However, the two pyridine rings in the title compound present a special angle, which may be of interest for the study of new functional caged compounds.

In the molecules forming the title crystal structure, the tricyclic structure formed by oxazinoquinoline structure is not in one plane. The dihedral angle of the phenyl ring formed by C23–28 and the phenyl ring formed by C6–C1 is 57°. The dihedral angle between the phenyl ring formed by C16–18 and the phenyl ring formed by C29–34 is 33.322 Å, close to a right angle. Several important bond angle data are involved as follows C2–C1–C6 = 121.9(2) Å, C36–O1–C35 = 117.56(13) Å, C16–N1–C14 = 120.10(11) Å. The bond lengths and angles are in the expected ranges [9, 10].

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