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Crystal structure of dimethyl (3aS,6R,6aS,7S)-2-pivaloyl-2,3-dihydro-1H,6H,7H-3a,6:7,9a-diepoxybenzo[de]isoquinoline-3a1,6a-dicarboxylate, $C_{21}H_{25}NO_8$

Abstract

$C_{21}H_{25}NO_8$, $P2_1/n$, (no. 14), $a = 12.905(3)$ Å, $b = 13.370(3)$ Å, $c = 13.090(3)$ Å, $\beta = 118.19(3)^\circ$, $V = 1990.5(8)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0629$, $wR_{ref}(F^2) = 0.1966$, $T = 100(2)$ K.

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Table 1: Data collection and handling.

Crystal:	Parallelepiped
Size:	$0.20 \times 0.20 \times 0.10$ mm
Wavelength:	Synchrotron radiation (0.9699 Å)
μ :	0.25 mm^{-1}
Diffractometer, scan mode:	Synchrotron Radiation, ϕ -scan
$\theta_{\max}$, completeness:	38.5° , 97%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	21424, 4031, 0.074
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2857
$N(\text{param})_{\text{refined}}$:	277
Programs:	Olex2 [14]

The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

Synthesis and characterization by $^1\text{H}/^{13}\text{C}$ NMR, IR and HRMS methods was reported in [10, 11].

Experimental details

H atoms were located in the difference Fourier map, but refined with fixed individual displacement parameters, using a riding model with C–H distances of 0.95–1.0 Å (for aromatic rings), and C–H distances 0.99, 0.98 Å for methylene and methyl groups, with $U(\text{H})$ values of $1.2U_{\text{eq}}(\text{C}_{\text{Ar}}$ and $\text{CH}_2)$ and $1.5U_{\text{eq}}(\text{C})$ (for CH_3).

Comment

Weak interactions, such as hydrogen, halogen, chalcogen, pnictogen, tetrel and icosagen bonds were extensively used in the synthesis, catalysis, crystal engineering, drug delivery, etc. [1–5]. Among those, hydrogen bonding has turned out

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

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
O1	0.53895(17)	0.50371(13)	0.14659(14)	0.0272(5)
O2	0.66863(17)	0.39465(12)	0.32594(14)	0.0272(5)
O3	0.41410(18)	0.34009(12)	0.36896(15)	0.0320(5)
O4	0.30855(18)	0.42559(13)	0.19913(15)	0.0327(5)
O5	0.92773(19)	0.51245(14)	0.25445(17)	0.0359(5)
O6	0.86050(18)	0.66528(13)	0.26739(17)	0.0347(5)
O7	0.84057(18)	0.66491(13)	0.47755(16)	0.0360(6)
O8	0.65940(17)	0.73253(12)	0.38401(14)	0.0288(5)
C1	0.5929(3)	0.48200(19)	0.4394(2)	0.0269(7)
H1A	0.595130	0.420758	0.482900	0.032*
H1B	0.607045	0.540190	0.491118	0.032*
N2	0.4770(2)	0.49140(15)	0.33724(18)	0.0258(6)
C3	0.4601(3)	0.57971(18)	0.2638(2)	0.0276(7)
H3A	0.463968	0.640920	0.308017	0.033*
H3B	0.381142	0.576736	0.195366	0.033*
C3A	0.5521(3)	0.58553(18)	0.2237(2)	0.0248(6)
C4	0.5559(3)	0.67432(19)	0.1512(2)	0.0289(7)
H4	0.526204	0.739848	0.148393	0.035*
C5	0.6094(3)	0.6401(2)	0.0933(2)	0.0306(7)
H5	0.627273	0.676562	0.041309	0.037*
C6	0.6365(3)	0.5299(2)	0.1269(2)	0.0283(7)
H6	0.645004	0.487444	0.068482	0.034*
C6A	0.7430(3)	0.52286(18)	0.2548(2)	0.0249(6)
C7	0.7680(3)	0.41286(18)	0.3066(2)	0.0277(7)
H7	0.780470	0.361709	0.257709	0.033*
C8	0.8675(3)	0.42068(17)	0.4315(2)	0.0262(6)
H8	0.947277	0.401213	0.460299	0.031*
C9	0.8177(3)	0.46082(17)	0.4912(2)	0.0249(6)
H9	0.854880	0.476216	0.571544	0.030*
C9A	0.6887(3)	0.47681(17)	0.4033(2)	0.0235(6)
C9B	0.6833(2)	0.56946(17)	0.3239(2)	0.0234(6)
C10	0.4013(3)	0.41226(18)	0.3062(2)	0.0253(6)
C11	0.2027(3)	0.3614(2)	0.1565(2)	0.0298(7)
C12	0.2313(3)	0.2527(2)	0.1461(3)	0.0422(8)
H12A	0.272911	0.248818	0.099988	0.063*
H12B	0.158281	0.214061	0.107969	0.063*
H12C	0.281257	0.225310	0.223532	0.063*
C13	0.1456(3)	0.3743(2)	0.2347(3)	0.0371(8)
H13A	0.199281	0.349841	0.312616	0.056*
H13B	0.072150	0.336168	0.202910	0.056*
H13C	0.128905	0.445317	0.238635	0.056*
C14	0.1251(3)	0.4053(3)	0.0365(3)	0.0463(9)
H14A	0.108058	0.475482	0.044112	0.069*
H14B	0.051330	0.367574	−0.001534	0.069*
H14C	0.166119	0.400875	−0.010054	0.069*
C15	0.8531(3)	0.56548(19)	0.2600(2)	0.0280(7)
C16	0.9690(3)	0.7089(2)	0.2794(3)	0.0386(8)
H16A	0.969886	0.705079	0.205033	0.058*
H16B	1.036385	0.672011	0.338315	0.058*
H16C	0.973921	0.779088	0.302933	0.058*
C17	0.7392(3)	0.65990(17)	0.4019(2)	0.0247(6)
C18	0.7011(3)	0.81678(19)	0.4637(2)	0.0343(7)
H18A	0.654694	0.876343	0.425467	0.051*
H18B	0.784086	0.829329	0.486658	0.051*
H18C	0.692729	0.801415	0.532610	0.051*

to be particularly suitable for design of organic and coordination compounds [6–9]. There is one title molecule in the asymmetric unit.

The molecule comprises a fused hexacyclic system containing four five-membered rings in the usual envelope conformations with Cremer and Pople parameters [12] ranging from $Q(2) = 0.517(3)–0.583(3)$ Å; $\varphi_2 = 175.2(3)–181.4(4)^\circ$ and one six-membered rings adopting a chair conformation with Cremer and Pople parameters $Q = 0.523(3)$ Å, $\theta = 6.8(3)^\circ$, $\varphi = 52(3)^\circ$ respectively. In the crystal structure the molecules are linked at least by C—O...H intermolecular hydrogen bond which link molecules into centrosymmetric dimers with $R_2^2(10)$ graph-set notation [13]. Other weak intermolecular interactions will contribute to the stability of the packing. The carboxylate groups are α -oriented, meanwhile, the *tert*-butyl carboxylate group is β -oriented. All distances and angles are normal.

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