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Crystal structure of dimethyl (3*aS*,6*R*,6*aS*,7*S*)-1*H*, 3*H*,6*H*,7*H*-3*a*,6:7,9*a*-diepoxybenzo[*de*]isochromene-3*a*¹,6*a*-dicarboxylate, C₁₆H₁₆O₇

Abstract

C₁₆H₁₆O₇, monoclinic, *P*₂/n (no. 14), *a* = 12.830(3) Å, *b* = 8.1101(16) Å, *c* = 14.900(3) Å, β = 114.88(3)°, *V* = 1406.5(6) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0578, *wR*_{ref}(*F*²) = 0.1799, *T* = 100(2) K.

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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.

Table 1: Crystal collection and handling.

Crystal:	Prism, colourless
Size:	0.25 × 0.15 × 0.10 mm
Wavelength:	synchrotron radiation (λ = 0.9699 Å)
μ:	0.282 mm ^{−1}
Diffractometer, scan mode:	synchrotron source
θ _{max} , completeness:	38.5°, >97%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	13125, 3051, 0.0965
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2σ(<i>I</i> _{obs}), 2293
<i>N</i> (<i>param</i>) _{refined} :	211
Programs:	SHELX [1]

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Source of materials

Synthesis and their characterization by ¹H and ¹³C-NMR, IR and HRMS methods are reported [21, 22].

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*(*H*) values of 1.2*U*_{eq}(C_{Ar} and CH₂) and 1.5*U*_{eq}(C) (for CH₃).

Discussion

Weak interactions, such as hydrogen, halogen, chalcogen, pnictogen, tetrel and icosagen bonds were extensively used

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

Atom	x	y	z	U_{iso}^*/U_{eq}
C1	0.49596(18)	0.7828(3)	0.74001(16)	0.0197(5)
H1A	0.557699	0.735234	0.800019	0.024
H1B	0.532417	0.853153	0.706970	0.024
O2	0.42176(12)	0.88305(19)	0.76850(11)	0.0203(4)
C3	0.32894(19)	0.9602(3)	0.68657(17)	0.0197(5)
H3A	0.361011	1.033878	0.651500	0.024
H3B	0.283276	1.028560	0.711962	0.024
C3A	0.25123(18)	0.8339(3)	0.61457(15)	0.0156(5)
C4	0.14311(18)	0.8870(3)	0.52386(16)	0.0169(5)
H4	0.128009	0.991704	0.492368	0.020
C5	0.07493(18)	0.7548(3)	0.49870(16)	0.0175(5)
H5	0.001691	0.745159	0.444519	0.021
C6	0.13864(17)	0.6225(3)	0.57473(15)	0.0164(5)
H6	0.088478	0.535838	0.583817	0.020
C6A	0.24101(17)	0.5543(3)	0.55197(16)	0.0157(5)
C7	0.32232(18)	0.4288(3)	0.63400(16)	0.0180(5)
H7	0.282245	0.331857	0.646628	0.022
C8	0.42161(18)	0.3862(3)	0.60684(17)	0.0201(6)
H8	0.432604	0.286106	0.578856	0.024
C9	0.48936(19)	0.5183(3)	0.63049(16)	0.0197(5)
H9	0.559281	0.531507	0.623734	0.024
C9A	0.43087(18)	0.6452(3)	0.67109(16)	0.0175(5)
C9B	0.32100(17)	0.7098(3)	0.57792(15)	0.0150(5)
O3	0.20060(12)	0.72308(18)	0.66095(10)	0.0165(4)
O4	0.37938(12)	0.53564(18)	0.71785(11)	0.0176(4)
C10	0.20355(18)	0.4746(3)	0.45164(17)	0.0173(5)
O5	0.24502(13)	0.4945(2)	0.39265(12)	0.0242(4)
O6	0.11832(12)	0.36485(18)	0.43728(11)	0.0199(4)
C11	0.07643(19)	0.2713(3)	0.34575(16)	0.0214(5)
H11A	0.048904	0.347361	0.289415	0.032
H11B	0.013231	0.199667	0.342040	0.032
H11C	0.138885	0.203744	0.344019	0.032
C12	0.35819(18)	0.7856(3)	0.50283(15)	0.0158(5)
O7	0.45792(12)	0.80065(19)	0.51634(11)	0.0226(4)
O8	0.27094(12)	0.84531(19)	0.42136(11)	0.0207(4)
C13	0.3039(2)	0.9123(3)	0.34665(18)	0.0280(6)
H13A	0.235775	0.955633	0.291368	0.042
H13B	0.338625	0.825145	0.322623	0.042
H13C	0.359680	1.001489	0.375553	0.042

in the synthesis, catalysis, crystal engineering, drug delivery, etc. [2–12]. Among those, hydrogen bonding has turned out to be particularly suitable for design of organic and coordination compounds [13–20].

The molecule comprises a fused hexacyclic system containing four five-membered rings in the usual envelope conformations with Cremer and Pople parameters [23] ranging from $Q(2) = 0.521(2)–0.581(2)$ Å; $\varphi_2 = 177.6(2)–181.6(3)^\circ$ and one six-membered ring adopting a chair conformation with Cremer and Pople parameters $Q = 0.536(2)$ Å, $\theta = 4.1(2)^\circ$, $\varphi = 14(4)^\circ$, respectively. In the crystal structure the molecules are linked by one strong $C–O \cdots H$ intermolecular hydrogen bonds which links molecules into centrosymmetric dimers

with $R^2_2(10)$ graph-set notation [24]. The carboxylate groups are α -oriented. All distances and angles are normal.

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