Crystal structure of methyl 4a*SR*,5*RS*,12a*RS*-12a-hydroxy-1,2,3,4,4a,5,6,11,12,12a-decahydro-dibenzo[*a,e*][8]annulene-5-carboxylate, C₁₈H₂₄O₃

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Abstract

C₁₈H₂₄O₃, monoclinic, *P*12₁/*a*1 (No. 14), *a* = 5.905(1) Å, *b* = 31.840(4) Å, *c* = 8.743(2) Å, β = 104.50(1)°, *V* = 1591.5 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.058, *wR*_{ref}(*F*²) = 0.177, *T* = 293 K.

Source of material

The title compound was obtained by samarium diiodide induced reaction of methyl 2*RS*,2'*RS*-2-(2'-oxocyclohexyl)-3-(2-vinylphenyl)propanoate in 50% yield (purified by chromatography on silica gel, colourless crystals, mp 353 K – 354 K, recrystallized from hexane/ethyl acetate) [1].

Discussion

The crystal structure proves the constitution and configuration of the title compound. The bridge-head 10-hydroxy group and the 8-methoxycarbonyl group are *trans* positioned. The eight-membered and the six-membered rings are *trans* annulated. An additional interesting feature of the structure is the boat-like conformation of the central eight-membered ring. Bonding distances and bond angles show no remarkable deviations from average values.

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.29 × 0.5 × 1.15 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
μ :	0.80 cm ⁻¹
Diffractometer, scan mode:	Enraf-Nonius CAD4, $\omega/2\theta$
$2\theta_{\max}$:	49.94°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	3062, 2781
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 1956
<i>N</i> (<i>param</i>) _{refined} :	190
Programs:	SHELXS-97 [2], SHELXL-97 [3]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1)	4e	−0.6304	0.6064	0.2507	0.127
H(1A)	4e	0.1419	0.7210	0.5278	0.096
H(2)	4e	0.4830	0.7140	0.7237	0.121
H(3)	4e	0.5304	0.6559	0.8874	0.127
H(4)	4e	0.2417	0.6064	0.8515	0.107
H(7A)	4e	−0.2348	0.7041	0.4017	0.068
H(7B)	4e	−0.3664	0.6676	0.4627	0.068
H(8)	4e	−0.3763	0.6529	0.2033	0.055
H(9)	4e	−0.0308	0.5967	0.3706	0.056
H(11A)	4e	−0.4641	0.6192	0.5183	0.093
H(11B)	4e	−0.4828	0.5726	0.5687	0.093
H(12A)	4e	−0.2016	0.5981	0.7595	0.106
H(12B)	4e	−0.0702	0.5714	0.6603	0.106
H(13A)	4e	−0.1617	0.5261	0.4441	0.110
H(13B)	4e	−0.4290	0.5161	0.4119	0.110
H(14A)	4e	−0.4632	0.5144	0.1418	0.118
H(14B)	4e	−0.2682	0.4826	0.2265	0.118
H(15A)	4e	0.0233	0.5299	0.2112	0.100
H(15B)	4e	−0.1405	0.5246	0.0406	0.100
H(16A)	4e	−0.3312	0.5869	0.0644	0.075
H(16B)	4e	−0.0624	0.5959	0.0983	0.075
H(18A)	4e	−0.0491	0.7370	−0.0505	0.157
H(18B)	4e	0.1161	0.6977	−0.0195	0.157
H(18C)	4e	0.1436	0.7330	0.1089	0.157

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(1)	4e	−0.5940(3)	0.58154(7)	0.2616(3)	0.042(1)	0.086(1)	0.124(2)	−0.0086(9)	0.016(1)	−0.015(1)
O(2)	4e	0.1660(3)	0.66089(6)	0.2504(2)	0.041(1)	0.092(1)	0.076(1)	0.0007(9)	0.0150(8)	0.013(1)
O(3)	4e	−0.1311(3)	0.69595(6)	0.0987(2)	0.064(1)	0.069(1)	0.075(1)	0.0084(9)	0.0249(9)	0.028(1)
C(1)	4e	0.1615(6)	0.6974(1)	0.5924(4)	0.093(2)	0.085(2)	0.067(2)	−0.017(2)	0.027(2)	−0.029(2)
C(2)	4e	0.3669(7)	0.6937(2)	0.7104(5)	0.089(3)	0.134(3)	0.082(2)	−0.031(2)	0.025(2)	−0.056(3)
C(3)	4e	0.3947(7)	0.6592(2)	0.8070(5)	0.090(3)	0.150(4)	0.067(2)	0.005(3)	0.002(2)	−0.043(3)
C(4)	4e	0.2209(7)	0.6295(1)	0.7844(4)	0.099(3)	0.112(3)	0.051(2)	0.028(2)	0.009(2)	−0.010(2)
C(5)	4e	0.0130(5)	0.63252(9)	0.6647(3)	0.077(2)	0.076(2)	0.044(1)	0.017(2)	0.017(1)	−0.003(1)
C(6)	4e	−0.0170(5)	0.66764(8)	0.5658(3)	0.065(2)	0.065(2)	0.050(1)	0.005(1)	0.022(1)	−0.015(1)
C(7)	4e	−0.2263(4)	0.67455(8)	0.4291(3)	0.062(2)	0.052(1)	0.062(2)	0.014(1)	0.026(1)	0.007(1)
C(8)	4e	−0.2249(4)	0.64858(7)	0.2791(3)	0.036(1)	0.052(1)	0.047(1)	0.006(1)	0.0058(9)	0.006(1)
C(9)	4e	−0.1852(4)	0.60108(7)	0.2986(3)	0.035(1)	0.050(1)	0.053(1)	0.005(1)	0.007(1)	0.001(1)
C(10)	4e	−0.3665(4)	0.57771(8)	0.3675(3)	0.045(1)	0.057(2)	0.080(2)	−0.001(1)	0.022(1)	0.003(1)
C(11)	4e	−0.3841(5)	0.5924(1)	0.5311(4)	0.080(2)	0.079(2)	0.087(2)	−0.009(2)	0.045(2)	0.012(2)
C(12)	4e	−0.1575(7)	0.5973(1)	0.6599(4)	0.109(3)	0.094(2)	0.064(2)	0.006(2)	0.025(2)	0.024(2)
C(13)	4e	−0.3128(6)	0.5308(1)	0.3711(5)	0.091(2)	0.059(2)	0.135(3)	−0.002(2)	0.047(2)	0.013(2)
C(14)	4e	−0.3091(6)	0.5122(1)	0.2137(5)	0.085(2)	0.057(2)	0.151(4)	0.001(2)	0.028(2)	−0.024(2)
C(15)	4e	−0.1330(6)	0.5351(1)	0.1459(4)	0.069(2)	0.073(2)	0.108(2)	0.004(2)	0.024(2)	−0.032(2)
C(16)	4e	−0.1814(5)	0.58183(8)	0.1384(3)	0.055(2)	0.071(2)	0.061(2)	−0.000(1)	0.012(1)	−0.015(1)
C(17)	4e	−0.0404(4)	0.66795(7)	0.2096(3)	0.045(1)	0.053(1)	0.045(1)	0.002(1)	0.011(1)	−0.001(1)
C(18)	4e	0.0338(7)	0.7178(1)	0.0284(5)	0.111(3)	0.092(2)	0.132(3)	0.007(2)	0.071(3)	0.046(2)

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