Crystal structure of (1*S*,3*S*,5*R*,7*R*,8*R*)-1,4,4-trimethyl-8-carbomethoxy-8-hydroxy-10-oxatricyclo[5.3.0.0^{3,5}]decan-9-one, C₁₄H₂₀O₅

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Abstract

C₁₄H₂₀O₅, orthorhombic, *P*2₁2₁2₁ (No. 19), *a* = 7.428(2) Å, *b* = 10.898(2) Å, *c* = 16.906(3) Å, *V* = 1368.5 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.044, *wR*(*F*²) = 0.099, *T* = 293 K.

Source of material

Compound C₁₄H₂₀O₅ was prepared by Mn(OAc)₃-mediated condensation of potassium monomethyl malonate on (+)-Δ³-carene in acetic acid under oxygen atmosphere. Crystals were obtained from hexane/ether (50/50) solution.

Table 1. Data collection and handling.

Crystal:	colourless, hexagonal prism, size 0.15 × 0.18 × 0.31 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
μ:	0.98 cm ⁻¹
Diffractometer, scan mode:	Enraf-Nonius CAD4, ω/2θ
2θ _{max} :	60.24°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	2323, 2323
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2302
<i>N</i> (<i>param</i>) _{refined} :	200
Programs:	DATARED [2], SHELXS-86 [3], SHELXL-93 [4], ORTEPII [5]

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(81)	4 <i>a</i>	-1.298(1)	-0.2722(4)	-0.0734(4)	0.05
H(82)	4 <i>a</i>	-1.4976(3)	-0.2329(6)	-0.0566(5)	0.05
H(83)	4 <i>a</i>	-1.341(1)	-0.1819(3)	-0.0038(1)	0.05
H(91)	4 <i>a</i>	-1.5964(2)	-0.0151(3)	-0.0935(6)	0.05
H(92)	4 <i>a</i>	-1.452(1)	0.0747(4)	-0.1282(4)	0.05
H(93)	4 <i>a</i>	-1.4447(9)	0.0375(6)	-0.0386(2)	0.05
H(131)	4 <i>a</i>	-0.7201(7)	-0.2751(8)	-0.26672(7)	0.05
H(132)	4 <i>a</i>	-0.7902(3)	-0.3364(4)	-0.1887(5)	0.05
H(133)	4 <i>a</i>	-0.6777(5)	-0.2148(4)	-0.1846(5)	0.05
H(191)	4 <i>a</i>	-1.5617(6)	-0.1315(5)	-0.4176(5)	0.05
H(192)	4 <i>a</i>	-1.4620(2)	-0.051(1)	-0.4810(1)	0.05
H(193)	4 <i>a</i>	-1.5441(7)	0.0107(6)	-0.4055(5)	0.05
H(1)	4 <i>a</i>	-1.059(3)	-0.331(2)	-0.277(1)	0.05
H(21)	4 <i>a</i>	-1.283(3)	-0.304(2)	-0.1949(9)	0.05
H(22)	4 <i>a</i>	-1.358(3)	-0.263(2)	-0.277(1)	0.05
H(3)	4 <i>a</i>	-1.375(3)	-0.061(2)	-0.234(1)	0.05
H(4)	4 <i>a</i>	-1.137(3)	0.018(2)	-0.150(1)	0.05
H(51)	4 <i>a</i>	-1.036(3)	-0.225(2)	-0.115(1)	0.05
H(52)	4 <i>a</i>	-0.913(3)	-0.101(2)	-0.115(1)	0.05
H(15)	4 <i>a</i>	-0.900(3)	-0.234(2)	-0.438(1)	0.05

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

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
C(1)	4 <i>a</i>	-1.0846(1)	-0.24746(6)	-0.27213(4)	0.0274(3)	0.0236(3)	0.0239(3)	0.0000(3)	-0.0008(3)	0.0012(2)
C(2)	4 <i>a</i>	-1.2746(1)	-0.23855(7)	-0.23624(5)	0.0266(3)	0.0284(3)	0.0280(3)	-0.0066(3)	0.0015(3)	-0.0017(2)
C(3)	4 <i>a</i>	-1.3162(1)	-0.11529(8)	-0.19952(5)	0.0297(3)	0.0312(3)	0.0264(3)	0.0016(3)	0.0020(3)	-0.0012(2)
C(4)	4 <i>a</i>	-1.1704(1)	-0.06585(8)	-0.14554(5)	0.0352(4)	0.0342(4)	0.0276(3)	-0.0068(3)	0.0038(3)	-0.0035(3)
C(5)	4 <i>a</i>	-1.0072(1)	-0.1468(1)	-0.13897(5)	0.0308(4)	0.0492(4)	0.0252(3)	-0.0049(4)	-0.0036(3)	-0.0016(3)
C(6)	4 <i>a</i>	-0.9379(1)	-0.18525(8)	-0.22009(5)	0.0246(3)	0.0335(3)	0.0265(3)	-0.0024(3)	-0.0006(3)	0.0025(3)

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Table 3. Continued.

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(7)	4a	-1.3530(1)	-0.09905(9)	-0.11157(5)	0.0351(4)	0.0413(4)	0.0275(3)	-0.0031(3)	0.0079(3)	-0.0049(3)
C(8)	4a	-1.3745(2)	-0.2062(1)	-0.05634(6)	0.0508(5)	0.0604(6)	0.0326(4)	-0.0098(5)	0.0097(4)	0.0070(4)
C(9)	4a	-1.4724(2)	0.0095(1)	-0.09110(9)	0.0533(6)	0.0525(5)	0.0510(6)	0.0048(5)	0.0146(5)	-0.0160(5)
C(10)	4a	-1.0517(1)	-0.18909(6)	-0.35409(4)	0.0282(3)	0.0264(3)	0.0242(3)	0.0009(3)	0.0020(2)	-0.0020(2)
C(11)	4a	-0.9604(1)	-0.06606(7)	-0.33521(5)	0.0298(3)	0.0309(3)	0.0304(3)	-0.0040(3)	0.0019(3)	-0.0004(3)
O(12)	4a	-0.8943(1)	-0.06938(6)	-0.26162(4)	0.0357(3)	0.0353(3)	0.0312(3)	-0.0098(3)	-0.0006(2)	-0.0007(2)
C(13)	4a	-0.7656(1)	-0.2597(1)	-0.21452(7)	0.0270(3)	0.0585(6)	0.0454(5)	0.0056(4)	-0.0064(4)	0.0006(4)
O(14)	4a	-0.9362(2)	0.01850(7)	-0.37858(5)	0.0617(5)	0.0404(3)	0.0441(4)	-0.0123(4)	0.0006(4)	0.0092(3)
O(15)	4a	-0.9219(1)	-0.26249(7)	-0.39303(4)	0.0401(3)	0.0426(3)	0.0300(3)	0.0108(3)	0.0093(3)	-0.0021(2)
C(16)	4a	-1.2209(1)	-0.17591(7)	-0.40500(4)	0.0347(4)	0.0339(3)	0.0228(3)	-0.0006(3)	-0.0013(3)	0.0033(3)
O(17)	4a	-1.2577(1)	-0.24655(8)	-0.45676(5)	0.0573(4)	0.0498(4)	0.0388(3)	0.0028(4)	-0.0168(3)	-0.0106(3)
O(18)	4a	-1.3172(1)	-0.07962(8)	-0.38400(5)	0.0404(4)	0.0489(4)	0.0413(3)	0.0130(3)	-0.0101(3)	-0.0057(3)
C(19)	4a	-1.4854(2)	-0.0614(2)	-0.4256(1)	0.0469(6)	0.0729(8)	0.0689(8)	0.0176(6)	-0.0205(6)	-0.0031(7)

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