Crystal structure of (2*R*,3*S*)-2*exo*-[1*R*-(2*S*-isopropyl-5*R*-methylcyclohexyloxy)]-2*endo*,3*exo*-dihydroxymethyl-bicyclo[2.2.1]hept-5-ene, (CH₂OH)₂C₇H₇OC₆H₉(CH₃)(C₃H₇)

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Abstract

C₁₉H₃₂O₃, orthorhombic, *P*2₁2₁2₁ (No. 19), *a* = 7.511(2) Å, *b* = 9.557(1) Å, *c* = 24.411(5) Å, *V* = 1752.3 Å³, *Z* = 4, *R*_g(*F*) = 0.052, *wR*_{ref}(*F*²) = 0.133, *T* = 293 K.

Source of material

The title compound was prepared by adding a solution of (2*R*,3*S*)-2-[1*R*-(2*S*-isopropyl-5*R*-methylcyclohexyloxy)]-2,3-dicarboxy-[1*R*-(2*S*-isopropyl-5*R*-methylcyclohexyl)bicyclo[2.2.1]hept-5-ene in THF to a suspension of LiAlH₄ in THF under argon atmosphere at 273 K. Recrystallization from hexane yielded colorless prisms.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.3 × 0.45 × 0.8 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.77 cm ⁻¹
Diffractometer, scan mode:	Bruker AXS P4, ω
2 θ _{max} :	55°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	5719, 4025
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 3095
<i>N</i> (<i>param</i>) _{refined} :	207
Program:	SHELXTL-PLUS [1]

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)	4 <i>a</i>	0.0746	0.2506	0.7933	0.08
H(3)	4 <i>a</i>	0.0001	0.0004	0.9194	0.08
H(4)	4 <i>a</i>	-0.3140	0.0505	0.8785	0.08
H(5)	4 <i>a</i>	-0.1575	-0.1215	0.8176	0.08
H(6)	4 <i>a</i>	0.0784	-0.0053	0.7694	0.08
H(7A)	4 <i>a</i>	-0.2517	0.2126	0.8002	0.08
H(7B)	4 <i>a</i>	-0.1926	0.2914	0.8548	0.08
H(8A)	4 <i>a</i>	0.2674	0.0042	0.8668	0.08
H(8B)	4 <i>a</i>	0.3656	0.1465	0.8561	0.08
H(8)	4 <i>a</i>	0.385(5)	0.170(3)	0.942(1)	0.07(1)
H(9A)	4 <i>a</i>	-0.1574	0.2581	0.9498	0.08
H(9B)	4 <i>a</i>	-0.2282	0.1138	0.9710	0.08
H(9)	4 <i>a</i>	0.095(9)	0.183(6)	0.990(2)	0.18(2)
H(11)	4 <i>a</i>	0.0698	0.4508	0.8462	0.08
H(12)	4 <i>a</i>	0.2750	0.5369	0.9376	0.08
H(13A)	4 <i>a</i>	0.1079	0.7092	0.8586	0.08
H(13B)	4 <i>a</i>	0.2126	0.7671	0.9094	0.08
H(14A)	4 <i>a</i>	0.3926	0.7717	0.8304	0.08
H(14B)	4 <i>a</i>	0.4782	0.6764	0.8757	0.08
H(15)	4 <i>a</i>	0.2815	0.5775	0.7851	0.08
H(16A)	4 <i>a</i>	0.3387	0.3493	0.8157	0.08
H(16B)	4 <i>a</i>	0.4379	0.4080	0.8674	0.08
H(17)	4 <i>a</i>	-0.0023	0.4679	0.9692	0.08
H(18A)	4 <i>a</i>	-0.1824	0.5205	0.8939	0.08
H(18B)	4 <i>a</i>	-0.1643	0.6804	0.9071	0.08
H(18C)	4 <i>a</i>	-0.2630	0.5811	0.9482	0.08
H(19A)	4 <i>a</i>	0.1464	0.6461	1.0166	0.08
H(19B)	4 <i>a</i>	-0.0603	0.6585	1.0239	0.08
H(19C)	4 <i>a</i>	0.0396	0.7585	0.9833	0.08
H(20A)	4 <i>a</i>	0.5707	0.6400	0.7596	0.08
H(20B)	4 <i>a</i>	0.5413	0.4787	0.7523	0.08
H(20C)	4 <i>a</i>	0.6482	0.5352	0.8028	0.08

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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)	4a	0.0198(3)	0.1825(2)	0.81808(8)	0.054(1)	0.039(1)	0.0363(9)	0.003(1)	−0.008(1)	−0.0018(8)
C(2)	4a	0.1021(3)	0.1744(2)	0.87687(8)	0.038(1)	0.0311(9)	0.0349(9)	0.0027(8)	0.0012(8)	−0.0004(8)
C(3)	4a	−0.0466(3)	0.0923(2)	0.90890(8)	0.037(1)	0.037(1)	0.044(1)	−0.0025(8)	−0.0003(9)	−0.0017(9)
C(4)	4a	−0.1946(3)	0.0717(2)	0.86462(9)	0.039(1)	0.047(1)	0.059(1)	−0.001(1)	−0.003(1)	−0.006(1)
C(5)	4a	−0.1197(4)	−0.0300(2)	0.8232(1)	0.057(2)	0.045(1)	0.059(1)	0.001(1)	−0.015(1)	−0.012(1)
C(6)	4a	0.0076(4)	0.0337(2)	0.79665(9)	0.059(2)	0.048(1)	0.045(1)	0.005(1)	−0.007(1)	−0.012(1)
C(7)	4a	−0.1754(3)	0.2089(2)	0.83228(9)	0.049(1)	0.050(1)	0.052(1)	0.010(1)	−0.015(1)	−0.010(1)
C(8)	4a	0.2812(3)	0.0995(2)	0.87986(8)	0.041(1)	0.039(1)	0.045(1)	0.0059(9)	0.002(1)	0.0006(9)
O(8)	4a	0.3487(3)	0.0973(2)	0.93365(8)	0.066(1)	0.062(1)	0.061(1)	0.013(1)	−0.0242(9)	0.0021(9)
C(9)	4a	−0.1222(3)	0.1636(3)	0.95939(9)	0.043(1)	0.054(1)	0.047(1)	−0.005(1)	0.007(1)	0.001(1)
O(9)	4a	−0.0007(3)	0.1695(2)	1.00387(7)	0.061(1)	0.093(1)	0.0410(8)	−0.005(1)	0.0037(9)	−0.0041(9)
O(10)	4a	0.1222(2)	0.3086(1)	0.90314(5)	0.0452(8)	0.0304(6)	0.0318(6)	−0.0008(6)	0.0021(6)	−0.0022(5)
C(11)	4a	0.1655(3)	0.4326(2)	0.87258(7)	0.040(1)	0.0319(9)	0.0327(8)	0.0025(8)	−0.0007(8)	0.0032(8)
C(12)	4a	0.1712(3)	0.5534(2)	0.91409(8)	0.037(1)	0.0337(9)	0.0367(9)	−0.0003(8)	−0.0018(8)	−0.0025(8)
C(13)	4a	0.2063(3)	0.6907(2)	0.88326(9)	0.055(1)	0.033(1)	0.055(1)	0.0017(9)	0.001(1)	0.003(1)
C(14)	4a	0.3784(4)	0.6852(2)	0.85068(9)	0.057(1)	0.041(1)	0.054(1)	−0.007(1)	0.008(1)	0.008(1)
C(15)	4a	0.3790(3)	0.5630(2)	0.81114(8)	0.051(1)	0.050(1)	0.038(1)	−0.002(1)	0.001(1)	0.0105(9)
C(16)	4a	0.3421(3)	0.4260(2)	0.84167(8)	0.047(1)	0.043(1)	0.0351(9)	0.001(1)	0.0042(9)	−0.0006(9)
C(17)	4a	0.0087(3)	0.5601(2)	0.95202(8)	0.045(1)	0.038(1)	0.044(1)	0.004(1)	0.005(1)	−0.0068(8)
C(18)	4a	−0.1665(3)	0.5881(3)	0.9226(1)	0.047(1)	0.067(2)	0.072(2)	0.010(1)	0.004(1)	−0.019(1)
C(19)	4a	0.0362(4)	0.6656(2)	0.99827(9)	0.068(2)	0.050(1)	0.053(1)	−0.001(1)	0.008(1)	−0.013(1)
C(20)	4a	0.5506(4)	0.5533(3)	0.7785(1)	0.072(2)	0.071(2)	0.055(1)	−0.008(2)	0.020(1)	0.010(1)

Reference

1. Sheldrick, G. M.: SHELXTL Release 5.1. Bruker Analytical X-ray Systems, 1997.