Crystal structure of 5-benzyloxy-3-hydroxy-10-methyl-tricyclo[3.6.0.0^{6,10}]-2,11-dioxaundecene(8), (C₇H₇O)(OH)(CH₃)C₉H₉O₂

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Source of material: The title compound was prepared by ozonation of 4-allyl-4-benzyloxy-3-hydroxy-1-methyl-2-oxa-8-(phenylselenyl)bicyclo[3.3.0]octane (see ref. 1). Subsequent treatment with dimethylsulfide at 193 K and the reaction with triethylamine at 273 K yielded the title compound.

C₁₇H₂₀O₄, orthorhombic, *Pbca* (No. 61), *a* = 7.154(1) Å, *b* = 16.514(2) Å, *c* = 25.425(2) Å, *V* = 3003.7 Å³, *Z* = 8, *R*(*F*) = 0.075, *R*_w(*F*) = 0.076.

Table 1. Parameters used for the X-ray data collection

Crystal:	colorless prism, size 0.3 x 0.3 x 0.25 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.90 cm ⁻¹
Diffractometer:	Siemens P4
Scan mode:	ω
<i>T</i> _{measurement} :	293 K
$2\theta_{\max}$:	55°
<i>N</i> (<i>hkl</i>) _{unique} :	3437
Criterion for <i>F</i> _o :	<i>F</i> _o > 3 σ (<i>F</i> _o)
<i>N</i> (<i>param</i>) _{refined} :	194
Program:	SHELXTL-plus

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1)	8c	0.4523(4)	0.2462(2)	0.1155(1)	0.08
H(3A)	8c	0.2974(5)	0.3131(2)	-0.0209(1)	0.08
H(3)	8c	0.594(5)	0.328(2)	-0.024(1)	0.09(1)
H(4A)	8c	0.2906(5)	0.4380(2)	0.0390(1)	0.08
H(4B)	8c	0.1294(5)	0.3738(2)	0.0411(1)	0.08
H(6)	8c	0.0883(4)	0.4039(2)	0.1382(1)	0.08
H(7A)	8c	0.1749(6)	0.4036(2)	0.2197(1)	0.08
H(7B)	8c	0.3676(6)	0.3672(2)	0.2022(1)	0.08
H(8)	8c	0.2029(5)	0.2653(3)	0.2588(1)	0.08
H(9)	8c	0.0540(5)	0.1794(2)	0.1962(1)	0.08
H(12A)	8c	0.4411(6)	0.5020(2)	0.0891(2)	0.08
H(12B)	8c	0.4047(6)	0.4935(2)	0.1496(2)	0.08
H(14)	8c	0.7626(6)	0.5115(3)	0.0577(2)	0.08
H(15)	8c	1.0511(8)	0.5690(4)	0.0710(3)	0.08
H(16)	8c	1.125(1)	0.6051(4)	0.1527(4)	0.08
H(17)	8c	0.951(2)	0.5843(5)	0.2238(4)	0.08
H(18)	8c	0.6514(9)	0.5231(3)	0.2108(2)	0.08
H(19A)	8c	-0.2033(4)	0.3023(2)	0.1439(1)	0.08
H(19B)	8c	-0.1720(4)	0.2192(2)	0.1154(1)	0.08
H(19C)	8c	-0.1385(4)	0.3005(2)	0.0850(1)	0.08

Table 3. Final 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)	8c	0.3608(4)	0.2679(2)	0.0917(1)	0.045(2)	0.045(2)	0.063(2)	0.002(1)	0.002(2)	-0.005(1)
O(2)	8c	0.4252(3)	0.2577(1)	0.04051(9)	0.077(2)	0.056(1)	0.079(2)	0.015(1)	0.026(1)	-0.002(1)
C(3)	8c	0.3716(5)	0.3272(2)	0.0091(1)	0.073(2)	0.069(2)	0.064(2)	0.007(2)	0.020(2)	0.005(2)
O(3)	8c	0.5306(4)	0.3658(2)	-0.0095(1)	0.096(2)	0.070(2)	0.091(2)	0.006(2)	0.038(2)	0.005(1)
C(4)	8c	0.2612(5)	0.3822(2)	0.0455(1)	0.068(2)	0.060(2)	0.070(2)	0.013(2)	0.009(2)	0.007(2)
C(5)	8c	0.3214(4)	0.3576(2)	0.1011(1)	0.043(2)	0.045(2)	0.063(2)	0.003(1)	0.005(2)	0.001(1)

Table 3. (Continued)

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(5)	8c	0.4934(3)	0.3941(1)	0.11666(9)	0.048(1)	0.043(1)	0.096(2)	−0.001(1)	0.002(1)	−0.008(1)
C(6)	8c	0.1661(4)	0.3571(2)	0.1433(1)	0.052(2)	0.051(2)	0.055(2)	0.004(2)	−0.001(2)	−0.009(1)
C(7)	8c	0.2344(6)	0.3602(2)	0.2009(1)	0.075(2)	0.102(3)	0.072(3)	−0.010(2)	−0.003(2)	−0.029(2)
C(8)	8c	0.1793(5)	0.2807(3)	0.2230(1)	0.072(2)	0.111(3)	0.059(2)	0.005(2)	−0.004(2)	0.017(2)
C(9)	8c	0.0960(5)	0.2333(2)	0.1887(1)	0.060(2)	0.070(2)	0.069(2)	0.004(2)	0.001(2)	0.010(2)
C(10)	8c	0.0723(4)	0.2736(2)	0.1364(1)	0.042(2)	0.053(2)	0.055(2)	0.004(1)	−0.000(1)	−0.000(1)
O(11)	8c	0.1823(3)	0.2304(1)	0.09660(8)	0.044(1)	0.051(1)	0.067(1)	−0.001(1)	−0.001(1)	−0.013(1)
C(12)	8c	0.4872(6)	0.4792(2)	0.1213(2)	0.067(2)	0.051(2)	0.254(7)	−0.002(2)	−0.009(4)	−0.009(3)
C(13)	8c	0.6766(5)	0.5123(2)	0.1326(2)	0.059(2)	0.041(2)	0.090(3)	−0.003(2)	−0.003(2)	0.003(2)
C(14)	8c	0.7983(6)	0.5265(3)	0.0928(2)	0.088(3)	0.084(3)	0.082(3)	0.023(3)	0.010(3)	0.014(2)
C(15)	8c	0.9674(8)	0.5601(4)	0.1000(3)	0.077(4)	0.093(4)	0.266(9)	0.022(3)	0.068(5)	0.092(5)
C(16)	8c	1.006(1)	0.5794(4)	0.1487(4)	0.122(6)	0.040(2)	0.42(2)	−0.001(3)	−0.14(1)	−0.004(7)
C(17)	8c	0.904(2)	0.5683(5)	0.1900(4)	0.31(2)	0.125(7)	0.188(8)	0.092(8)	−0.18(1)	−0.080(6)
C(18)	8c	0.7310(9)	0.5328(3)	0.1811(2)	0.175(6)	0.102(4)	0.070(3)	0.046(4)	0.003(3)	−0.006(3)
C(19)	8c	−0.1285(4)	0.2740(2)	0.1185(1)	0.047(2)	0.080(2)	0.082(2)	0.003(2)	−0.004(2)	−0.010(2)

References

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2. Sheldrick, G. M.: Program Package SHELXTL-plus. Release 4.1. Siemens Analytical X-Ray Instruments Inc., Madison (WI 53719), USA 1990.