Crystal structure of (2*S*,5*S*,10*S*)-2-benzyl-1-aza-4-oxatricyclo[8.3.0.0^{5,10}]tridecan-7,13-dione, C₁₁H₁₄NO₃(CH₂C₆H₅)

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Abstract

C₁₈H₂₁NO₃, orthorhombic, *P*2₁2₁2₁ (No. 19), *a* = 7.689(1) Å, *b* = 17.711(2) Å, *c* = 23.164(2) Å, *V* = 3154.5 Å³, *Z* = 8, *R*_g(*F*) = 0.070, *wR*(*F*) = 0.052, *T* = 293 K.

Source of material

The title compound was formed in 96% yield from (2*S*,5*S*,10*R*)-2-benzyl-1-aza-4-oxatricyclo[8.3.0.0^{5,10}]tridecan-8-en-7,13-dione (**I**) by catalytic hydrogenation [10% Pd(C)] of the enone system in ethyl acetate at atmospheric pressure. The α, β-unsaturated ketone **I** was obtained as a single diastereomer (*de* > 95 %) through spontaneous intramolecular Michael-type cyclization (donor: hydroxyl group - acceptor: dienone) of 1-[(*S*)-1'-benzyl-2'-hydroxyethyl]-1-azaspiro[4.5]deca-6,9-dien-2,8-dione [1].

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.2 × 0.25 × 0.45 mm
Wavelength:	Mo <i>K</i> α radiation (0.71073 Å)
μ:	0.90 cm ⁻¹
Diffractionmeter, scan mode:	Bruker AXS P4, ω
2θ _{max} :	55°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	9641, 7204
Criterion for <i>F</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>F</i> _{obs} > 3 σ(<i>F</i> _{obs}), 4003
<i>N</i> (<i>param</i>) _{refined} :	397
Program:	SHELXTL-plus [2]

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(12A)	4a	−0.1490(5)	0.8471(3)	0.3820(2)	0.08
H(12B)	4a	−0.1455(5)	0.9346(3)	0.3925(2)	0.08
H(13A)	4a	−0.3212(5)	0.8275(3)	0.4581(2)	0.08
H(13B)	4a	−0.3742(5)	0.9122(3)	0.4492(2)	0.08
H(16)	4a	0.0511(5)	0.9550(2)	0.5746(1)	0.08
H(17A)	4a	0.3499(6)	0.9714(2)	0.5445(2)	0.08
H(17B)	4a	0.2127(6)	1.0179(2)	0.5093(2)	0.08
H(19)	4a	0.1215(5)	0.9800(2)	0.4183(1)	0.08
H(11A)	4a	0.1793(5)	0.9169(2)	0.3359(2)	0.08
H(11B)	4a	0.3614(5)	0.9384(2)	0.3615(2)	0.08
H(11C)	4a	0.0855(6)	0.7713(2)	0.3580(2)	0.08
H(11D)	4a	0.2296(6)	0.7229(2)	0.3883(2)	0.08
H(11E)	4a	0.1944(5)	0.7928(2)	0.4720(1)	0.08
H(11F)	4a	0.0097(5)	0.7629(2)	0.4548(1)	0.08
H(11G)	4a	0.2662(5)	0.8315(2)	0.5631(1)	0.08
H(11H)	4a	0.0810(5)	0.8212(2)	0.5903(1)	0.08
H(116)	4a	0.0051(6)	0.8907(3)	0.6794(2)	0.08
H(117)	4a	0.1063(9)	0.9412(3)	0.7671(2)	0.08
H(118)	4a	0.403(1)	0.9623(3)	0.7805(2)	0.08
H(119)	4a	0.5955(8)	0.9385(3)	0.7061(2)	0.08
H(120)	4a	0.4975(6)	0.8813(3)	0.6213(2)	0.08
H(22A)	4a	−0.1503(5)	0.6519(3)	0.7726(2)	0.08
H(22B)	4a	−0.1561(5)	0.7333(3)	0.7990(2)	0.08
H(23A)	4a	−0.3764(6)	0.6871(3)	0.8508(2)	0.08
H(23B)	4a	−0.3056(6)	0.6043(3)	0.8460(2)	0.08
H(26)	4a	0.0635(5)	0.7048(2)	0.9776(1)	0.08
H(27A)	4a	0.3577(5)	0.7320(2)	0.9472(2)	0.08
H(27B)	4a	0.2148(5)	0.7869(2)	0.9235(2)	0.08
H(29)	4a	0.1164(6)	0.7770(2)	0.8294(1)	0.08
H(21A)	4a	0.3431(6)	0.7585(3)	0.7603(2)	0.08
H(21B)	4a	0.1585(6)	0.7353(3)	0.7376(2)	0.08
H(21C)	4a	0.0867(6)	0.5906(3)	0.7349(2)	0.08
H(21D)	4a	0.2356(6)	0.5359(3)	0.7546(2)	0.08
H(21E)	4a	0.2054(5)	0.5793(2)	0.8492(1)	0.08
H(21F)	4a	0.0212(5)	0.5522(2)	0.8288(1)	0.08
H(21G)	4a	0.2939(6)	0.5952(2)	0.9446(1)	0.08
H(21H)	4a	0.1050(6)	0.5683(2)	0.9597(1)	0.08
H(216)	4a	−0.0128(6)	0.6312(2)	1.0560(2)	0.08
H(217)	4a	0.0456(8)	0.6329(3)	1.1550(2)	0.08
H(218)	4a	0.3247(9)	0.6058(3)	1.1879(2)	0.08
H(219)	4a	0.5454(7)	0.5811(3)	1.1227(2)	0.08
H(220)	4a	0.4904(6)	0.5862(2)	1.0219(2)	0.08

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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
C(11)	4a	0.0337(5)	0.8774(2)	0.4436(2)	0.034(2)	0.059(3)	0.044(2)	−0.001(2)	−0.004(2)	0.008(2)
C(12)	4a	−0.1387(5)	0.8860(3)	0.4107(2)	0.036(2)	0.080(3)	0.061(2)	−0.003(2)	−0.008(2)	0.014(2)
C(13)	4a	−0.2791(5)	0.8785(3)	0.4568(2)	0.036(3)	0.067(3)	0.089(3)	−0.002(2)	−0.004(2)	0.007(3)
C(14)	4a	−0.1897(5)	0.8989(2)	0.5123(2)	0.041(3)	0.054(3)	0.066(3)	0.002(2)	0.006(2)	0.011(2)
O(14)	4a	−0.2577(4)	0.9127(2)	0.5590(1)	0.058(2)	0.095(3)	0.076(2)	0.007(2)	0.031(2)	0.006(2)
N(15)	4a	−0.0169(4)	0.9011(2)	0.5028(1)	0.032(2)	0.059(2)	0.048(2)	0.004(2)	0.001(2)	0.007(2)
C(16)	4a	0.1077(5)	0.9231(2)	0.5468(1)	0.048(2)	0.049(2)	0.035(2)	0.009(2)	0.005(2)	−0.002(2)
C(17)	4a	0.2535(6)	0.9681(2)	0.5183(2)	0.062(3)	0.051(3)	0.046(2)	−0.002(2)	0.004(2)	−0.001(2)
O(18)	4a	0.3114(3)	0.9329(1)	0.4661(1)	0.035(2)	0.053(2)	0.051(1)	−0.003(1)	−0.001(1)	0.001(1)
C(19)	4a	0.1765(5)	0.9319(2)	0.4238(1)	0.039(2)	0.051(2)	0.047(2)	0.003(2)	0.002(2)	0.008(2)
C(110)	4a	0.2595(5)	0.9079(2)	0.3669(2)	0.050(2)	0.061(3)	0.041(2)	−0.003(2)	0.003(2)	0.005(2)
C(111)	4a	0.3142(6)	0.8270(3)	0.3655(2)	0.051(3)	0.075(3)	0.034(2)	0.006(3)	−0.000(2)	−0.003(2)
O(111)	4a	0.4552(4)	0.8074(2)	0.3478(1)	0.069(2)	0.101(3)	0.074(2)	0.019(2)	0.027(2)	−0.001(2)
C(112)	4a	0.1787(6)	0.7722(2)	0.3855(2)	0.058(3)	0.057(3)	0.052(2)	0.006(3)	−0.006(2)	−0.004(2)
C(113)	4a	0.1028(5)	0.7961(2)	0.4440(1)	0.043(2)	0.056(3)	0.052(2)	−0.003(2)	−0.002(2)	0.004(2)
C(114)	4a	0.1741(5)	0.8563(2)	0.5837(1)	0.057(3)	0.049(3)	0.048(2)	0.008(2)	0.000(2)	0.002(2)
C(115)	4a	0.2413(6)	0.8836(2)	0.6413(2)	0.056(3)	0.043(2)	0.045(2)	0.005(2)	0.009(2)	0.004(2)
C(116)	4a	0.1266(6)	0.9004(3)	0.6854(2)	0.067(3)	0.073(3)	0.054(3)	0.017(3)	0.011(3)	0.012(2)
C(117)	4a	0.1880(9)	0.9305(3)	0.7369(2)	0.123(5)	0.094(4)	0.046(3)	0.044(4)	0.021(3)	0.004(3)
C(118)	4a	0.363(1)	0.9434(3)	0.7440(2)	0.134(6)	0.086(4)	0.065(3)	0.023(4)	−0.032(4)	−0.017(3)
C(119)	4a	0.4748(8)	0.9260(3)	0.7015(2)	0.083(4)	0.091(4)	0.102(4)	−0.010(4)	−0.032(4)	−0.017(4)
C(120)	4a	0.4158(6)	0.8965(3)	0.6504(2)	0.051(3)	0.080(3)	0.070(3)	0.000(3)	0.003(2)	−0.009(3)
C(21)	4a	0.0363(5)	0.6675(2)	0.8364(1)	0.038(2)	0.055(3)	0.035(2)	0.005(2)	0.001(2)	−0.008(2)
C(22)	4a	−0.1412(5)	0.6807(3)	0.8076(2)	0.050(3)	0.086(3)	0.048(2)	0.006(3)	−0.001(2)	−0.002(2)
C(23)	4a	−0.2741(6)	0.6561(3)	0.8523(2)	0.037(3)	0.108(4)	0.072(3)	0.007(3)	−0.002(2)	−0.005(3)
C(24)	4a	−0.1822(6)	0.6641(3)	0.9091(2)	0.051(3)	0.065(3)	0.057(3)	0.006(3)	0.002(2)	−0.008(2)
O(24)	4a	−0.2465(4)	0.6632(2)	0.9575(1)	0.059(2)	0.109(3)	0.059(2)	0.001(2)	0.020(2)	0.001(2)
N(25)	4a	−0.0104(4)	0.6730(2)	0.8983(1)	0.034(2)	0.061(2)	0.044(2)	−0.001(2)	0.001(2)	−0.003(2)
C(26)	4a	0.1171(5)	0.6829(2)	0.9442(1)	0.052(3)	0.053(3)	0.036(2)	0.003(2)	−0.003(2)	−0.009(2)
C(27)	4a	0.2580(5)	0.7361(2)	0.9225(2)	0.062(3)	0.059(3)	0.045(2)	−0.010(3)	−0.006(2)	−0.009(2)
O(28)	4a	0.3110(3)	0.7197(2)	0.8646(1)	0.045(2)	0.069(2)	0.051(1)	−0.008(2)	−0.002(1)	−0.005(1)
C(29)	4a	0.1706(6)	0.7286(2)	0.8248(1)	0.050(3)	0.052(3)	0.047(2)	0.003(2)	−0.001(2)	0.004(2)
C(210)	4a	0.2491(6)	0.7233(3)	0.7646(2)	0.063(3)	0.078(3)	0.048(2)	−0.002(3)	0.006(2)	0.011(2)
C(211)	4a	0.3121(7)	0.6459(3)	0.7507(2)	0.058(3)	0.099(4)	0.035(2)	0.017(3)	0.000(2)	−0.003(2)
O(211)	4a	0.4555(4)	0.6341(2)	0.7315(1)	0.069(2)	0.156(4)	0.070(2)	0.031(2)	0.024(2)	−0.003(2)
C(212)	4a	0.1820(6)	0.5841(3)	0.7612(2)	0.077(3)	0.066(3)	0.048(2)	0.026(3)	−0.007(2)	−0.014(2)
C(213)	4a	0.1107(5)	0.5893(2)	0.8233(1)	0.055(3)	0.058(3)	0.048(2)	0.001(2)	−0.003(2)	−0.008(2)
C(214)	4a	0.1908(6)	0.6067(2)	0.9663(1)	0.065(3)	0.054(3)	0.047(2)	0.007(2)	−0.004(2)	−0.005(2)
C(215)	4a	0.2329(5)	0.6084(2)	1.0296(2)	0.052(3)	0.042(2)	0.050(2)	0.002(2)	−0.003(2)	−0.006(2)
C(216)	4a	0.1034(6)	0.6218(2)	1.0693(2)	0.062(3)	0.061(3)	0.060(3)	0.006(3)	0.009(2)	0.008(2)
C(217)	4a	0.1370(8)	0.6224(3)	1.1279(2)	0.107(5)	0.081(4)	0.053(3)	0.010(4)	0.026(3)	0.011(3)
C(218)	4a	0.3023(9)	0.6062(3)	1.1471(2)	0.143(6)	0.066(3)	0.048(3)	0.000(4)	−0.018(4)	0.009(3)
C(219)	4a	0.4309(7)	0.5923(3)	1.1086(2)	0.081(4)	0.075(4)	0.076(3)	0.015(3)	−0.029(3)	0.002(3)
C(220)	4a	0.3982(6)	0.5937(2)	1.0493(2)	0.063(3)	0.065(3)	0.060(3)	0.015(3)	−0.006(3)	−0.003(2)

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