Crystal structure of (3*aS*,4*R*,5*S*,6*R*,6*aS*)-4,5,6-triacetoxy-1-benzyl-3,3*a*,4,5,6,6*a*-hexahydro-1*H*-cyclopent[*c*]-isoxazole, C₁₉H₂₃NO₇

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Abstract

C₁₉H₂₃NO₇, orthorhombic, *P*2₁2₁2₁ (No. 19), *a* = 8.297(4) Å, *b* = 9.266(4) Å, *c* = 25.17(1) Å, *V* = 1935.3 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.057, *R*_w(*F*²) = 0.160, *T* = 293 K.

Source of material

The title compound has been obtained by intramolecular nitron cycloaddition [1–3]. Starting from methyl 6-iodo-2,3,4-tri-*O*-acetyl-α-*D*-manno-pyranoside [4, 5], reaction with zinc and vitamin B₁₂ as catalyst afforded the corresponding 5-hexenose [5, 6], which was transformed into the title compound without further purification by addition of *N*-benzyl-hydroxylamine and pyridine as a base (d. r. > 95/5). Purification by flash chromatography on silica and crystallization from ethyl acetate gave the isoxazolidine, (mp 382 K–383 K, [α]_D²⁰ = –120, *c* = 0.87, CHCl₃).

Discussion

The packing diagramme shows antiparallel strangs along the *b*-axis.

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.5 x 0.75 x 1.25 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.99 cm ⁻¹
Diffractometer, scan mode:	Nicolet P3, Wyckoff
2θ _{max} :	55°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	2546, 2463
Criterion for <i>I</i> _{obs} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2235
<i>N</i> (<i>param</i>) _{refined} :	245
Programs:	SHELXS-86 [7], SHELXL-93 [8], SHELXTL-plus [9]

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(3A)	4 <i>a</i>	–0.2495(5)	0.4886(5)	0.7557(2)	0.076
H(3B)	4 <i>a</i>	–0.1546(5)	0.6161(5)	0.7284(2)	0.076
H(3A1)	4 <i>a</i>	–0.1977(4)	0.7208(4)	0.8087(1)	0.058
H(4)	4 <i>a</i>	–0.2505(3)	0.6050(3)	0.8851(1)	0.055
H(5)	4 <i>a</i>	–0.0225(3)	0.3853(3)	0.8607(1)	0.051
H(6)	4 <i>a</i>	0.1863(3)	0.5457(3)	0.8766(1)	0.051
H(6A)	4 <i>a</i>	0.0748(4)	0.7432(3)	0.8143(1)	0.050
H(9A)	4 <i>a</i>	0.0470(7)	0.9131(6)	0.9616(2)	0.126
H(9B)	4 <i>a</i>	0.2219(7)	0.9011(6)	0.9847(2)	0.126
H(9C)	4 <i>a</i>	0.0760(7)	0.8356(6)	1.0161(2)	0.126
H(12A)	4 <i>a</i>	–0.1032(6)	0.3528(6)	1.0230(2)	0.123
H(12B)	4 <i>a</i>	0.0718(6)	0.2919(6)	1.0300(2)	0.123
H(12C)	4 <i>a</i>	–0.0691(6)	0.1884(6)	1.0136(2)	0.123
H(15A)	4 <i>a</i>	–0.5020(4)	0.2350(4)	0.8378(2)	0.089
H(15B)	4 <i>a</i>	–0.6356(4)	0.3344(4)	0.8132(2)	0.089
H(15C)	4 <i>a</i>	–0.6374(4)	0.2996(4)	0.8742(2)	0.089
H(16A)	4 <i>a</i>	0.2626(5)	0.7035(4)	0.7479(1)	0.066
H(16B)	4 <i>a</i>	0.0989(5)	0.6912(4)	0.7171(1)	0.066
H(18)	4 <i>a</i>	0.4564(4)	0.4856(4)	0.7367(2)	0.068
H(19)	4 <i>a</i>	0.5897(5)	0.3591(5)	0.6712(2)	0.079
H(20)	4 <i>a</i>	0.4829(6)	0.3404(5)	0.5874(2)	0.084
H(21)	4 <i>a</i>	0.2342(6)	0.4447(5)	0.5697(1)	0.083
H(22)	4 <i>a</i>	0.1023(5)	0.5735(4)	0.6344(1)	0.072

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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
N(1)	4a	0.1171(3)	0.5491(3)	0.7774(1)	0.043(1)	0.039(1)	0.045(1)	−0.005(1)	0.003(1)	−0.002(1)
O(2)	4a	−0.0126(3)	0.4610(3)	0.7559(1)	0.056(1)	0.047(1)	0.058(1)	−0.012(1)	0.003(1)	−0.012(1)
C(3)	4a	−0.1534(5)	0.5479(5)	0.7576(2)	0.050(2)	0.080(3)	0.059(2)	−0.005(2)	−0.015(2)	−0.008(2)
C(3A)	4a	−0.1447(4)	0.6264(4)	0.8106(1)	0.037(1)	0.047(2)	0.062(2)	0.009(1)	−0.010(1)	−0.002(2)
C(4)	4a	−0.2028(3)	0.5397(3)	0.8588(1)	0.027(1)	0.045(2)	0.065(2)	0.002(1)	−0.001(1)	−0.013(1)
C(5)	4a	−0.0505(3)	0.4707(3)	0.8817(1)	0.032(1)	0.046(1)	0.050(1)	0.006(1)	0.004(1)	0.001(1)
C(6)	4a	0.0771(3)	0.5853(3)	0.8739(1)	0.030(1)	0.052(2)	0.046(1)	0.003(1)	−0.001(1)	−0.004(1)
C(6A)	4a	0.0407(4)	0.6425(3)	0.8182(1)	0.039(1)	0.039(1)	0.046(1)	−0.001(1)	−0.002(1)	−0.002(1)
O(7)	4a	0.0527(3)	0.7061(3)	0.90920(9)	0.042(1)	0.061(1)	0.050(1)	0.001(1)	−0.008(1)	−0.013(1)
C(8)	4a	0.1423(5)	0.7128(5)	0.9530(1)	0.048(2)	0.097(3)	0.045(2)	−0.006(2)	−0.008(2)	−0.006(2)
O(8)	4a	0.2262(5)	0.6134(5)	0.9671(1)	0.100(3)	0.137(3)	0.070(2)	0.024(3)	−0.035(2)	−0.007(2)
C(9)	4a	0.1198(7)	0.8532(6)	0.9814(2)	0.086(3)	0.105(3)	0.061(2)	−0.016(3)	−0.011(2)	−0.025(2)
O(10)	4a	−0.0769(3)	0.4294(3)	0.93545(9)	0.050(1)	0.065(1)	0.052(1)	0.007(1)	0.013(1)	0.006(1)
C(11)	4a	0.0077(4)	0.3137(4)	0.9528(1)	0.043(2)	0.063(2)	0.059(2)	−0.004(2)	−0.003(2)	0.008(2)
O(11)	4a	0.0979(3)	0.2468(3)	0.9258(1)	0.064(2)	0.076(2)	0.065(1)	0.021(2)	0.003(1)	0.012(1)
C(12)	4a	−0.0262(6)	0.2841(6)	1.0099(2)	0.081(3)	0.102(4)	0.063(2)	0.003(3)	0.012(2)	0.021(2)
O(13)	4a	−0.3144(3)	0.4238(3)	0.8470(1)	0.030(1)	0.056(1)	0.101(2)	−0.003(1)	0.008(1)	−0.024(1)
C(14)	4a	−0.4678(4)	0.4459(4)	0.8542(2)	0.033(1)	0.052(2)	0.093(2)	0.001(2)	0.014(2)	−0.014(2)
O(14)	4a	−0.5160(4)	0.5596(5)	0.8631(4)	0.043(2)	0.096(3)	0.48(1)	−0.011(2)	0.031(4)	−0.127(5)
C(15)	4a	−0.5697(4)	0.3174(4)	0.8439(2)	0.041(2)	0.058(2)	0.078(2)	−0.004(2)	0.007(2)	−0.001(2)
C(16)	4a	0.1847(5)	0.6359(4)	0.7336(1)	0.066(2)	0.045(2)	0.054(2)	−0.009(2)	0.012(2)	0.002(1)
C(17)	4a	0.2651(4)	0.5445(3)	0.6920(1)	0.050(2)	0.043(1)	0.048(1)	−0.010(1)	0.006(1)	0.006(1)
C(18)	4a	0.4117(4)	0.4786(4)	0.7030(2)	0.053(2)	0.060(2)	0.056(2)	−0.004(2)	−0.005(2)	0.006(2)
C(19)	4a	0.4914(5)	0.4026(5)	0.6636(2)	0.055(2)	0.068(2)	0.075(2)	0.004(2)	0.006(2)	0.002(2)
C(20)	4a	0.4279(6)	0.3904(5)	0.6138(2)	0.074(3)	0.066(2)	0.070(2)	−0.006(2)	0.023(2)	−0.009(2)
C(21)	4a	0.2801(6)	0.4539(5)	0.6032(1)	0.087(3)	0.077(3)	0.044(2)	−0.015(3)	0.004(2)	−0.005(2)
C(22)	4a	0.2009(5)	0.5306(4)	0.6421(1)	0.059(2)	0.065(2)	0.055(2)	−0.005(2)	−0.004(2)	0.007(2)

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