Crystal structure of (3*aR*,4*R*,5*S*,6*S*,6*aR*)-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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carbons. All large components of the displacement ellipsoids are in-plane with the benzyl moiety, which indicates a strong in-plane vibration of the benzyl moiety. A discrete fixing of disorder positions is not useful, because this is a dynamical effect.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.50 × 0.65 × 0.75 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.98 cm ⁻¹
Diffractometer, scan mode:	Nicolet P3, Wyckoff
2 θ _{max} :	55°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	2562, 2562
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1760
$N(\text{param})_{\text{refined}}$:	245
Programs:	SHELXS-86 [6], SHELXL-93 [7]

Abstract

C₁₉H₂₃NO₇, orthorhombic, $P2_12_12_1$ (No. 19), $a = 9.854(3)$ Å, $b = 13.353(3)$ Å, $c = 14.949(3)$ Å, $V = 1967.0$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.059$, $wR_{\text{all}}(F^2) = 0.145$, $T = 293$ K.

Source of material

The title compound has been obtained by intramolecular nitron cycloaddition [1]. Starting from 6-deoxy-6-iodo-2,3,4-tri-*O*-acetyl- α -D-glucopyranoside [1–4], reaction with zinc and vitamin B₁₂ as a catalyst afforded the corresponding 5-hexenose [5], which was transformed into the title compound by addition of *N*-benzyl-hydroxylamine. Purification by flash chromatography on silica and crystallization from methanol gave the isoxazolidine as a single diastereomer in the form of colorless crystals [mp 381–383 K, $[\alpha]_{20}^D = -45$ ($c = 0.9$, CHCl₃)].

Discussion

The two five-membered rings of the bicyclic isoxazolidine structure each possess a twist-envelope conformation with O1 and C4 above the respective planes. The hydrogens of the C15-methyl-group show a rotational disorder with six equivalent hydrogen positions. In the benzyl-moiety we found large displacement parameters. They increase from ortho- over meta- to para-

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	Occ.	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1A)	4a		0.4477(4)	0.1110(4)	0.4683(4)	0.107
H(1B)	4a		0.4620(4)	0.1535(4)	0.5663(4)	0.107
H(2)	4a		0.2443(4)	0.1877(3)	0.4655(3)	0.073
H(3)	4a		0.2843(4)	0.2116(3)	0.6513(3)	0.067
H(4)	4a		0.0121(4)	0.1975(3)	0.5890(2)	0.061
H(5)	4a		0.1648(3)	0.0385(3)	0.6683(3)	0.061
H(6)	4a		0.1091(4)	0.0519(3)	0.4816(3)	0.066
H(7A)	4a		0.3802(5)	-0.0986(4)	0.4285(3)	0.106
H(7B)	4a		0.3145(5)	0.0017(4)	0.3951(3)	0.106
H(9)	4a		0.1346(8)	-0.0157(7)	0.2958(5)	0.163
H(10)	4a		-0.035(1)	-0.106(1)	0.231(1)	0.276
H(11)	4a		-0.091(2)	-0.257(1)	0.283(1)	0.293
H(12)	4a		0.0116(8)	-0.3207(7)	0.4084(7)	0.192
H(13)	4a		0.1868(6)	-0.2317(4)	0.4743(4)	0.118
H(15A)	4a	0.5	0.1721(5)	0.4921(3)	0.5552(3)	0.119
H(15B)	4a	0.5	0.3103(5)	0.5097(3)	0.5057(3)	0.119
H(15C)	4a	0.5	0.2998(5)	0.5278(3)	0.6090(3)	0.119
H(15D)	4a	0.5	0.3493(5)	0.5277(3)	0.5581(3)	0.119
H(15E)	4a	0.5	0.2111(5)	0.5100(3)	0.6076(3)	0.119
H(15F)	4a	0.5	0.2217(5)	0.4920(3)	0.5042(3)	0.119
H(17A)	4a		-0.0114(5)	0.2490(4)	0.8655(3)	0.130
H(17B)	4a		-0.1563(5)	0.2005(4)	0.8639(3)	0.130
H(17C)	4a		-0.1384(5)	0.3145(4)	0.8417(3)	0.130
H(19A)	4a		-0.2289(5)	-0.0719(4)	0.6230(3)	0.133
H(19B)	4a		-0.2713(5)	-0.0383(4)	0.7195(3)	0.133
H(19C)	4a		-0.2001(5)	-0.1420(4)	0.7049(3)	0.133

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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
O(1)	4a	0.3937(3)	0.0126(2)	0.5626(2)	0.056(2)	0.065(2)	0.130(3)	0.008(2)	0.000(2)	-0.003(2)
N(1)	4a	0.2702(3)	-0.0286(3)	0.5250(2)	0.055(2)	0.063(2)	0.082(2)	0.004(2)	0.011(2)	-0.005(2)
C(1)	4a	0.4066(4)	0.1124(4)	0.5273(4)	0.054(2)	0.069(3)	0.146(5)	0.002(2)	0.023(3)	-0.005(3)
O(2)	4a	0.1972(3)	0.3220(2)	0.5805(2)	0.051(1)	0.052(2)	0.081(2)	0.002(1)	0.002(2)	0.003(1)
C(2)	4a	0.2611(4)	0.1533(3)	0.5223(3)	0.051(2)	0.056(2)	0.076(3)	0.006(2)	0.017(2)	0.005(2)
O(3)	4a	0.4199(3)	0.3490(2)	0.5861(3)	0.053(2)	0.076(2)	0.136(3)	-0.011(2)	0.001(2)	0.016(2)
C(3)	4a	0.2183(4)	0.2180(3)	0.6026(3)	0.047(2)	0.047(2)	0.072(2)	0.003(2)	0.002(2)	0.004(2)
O(4)	4a	0.0498(3)	0.2054(2)	0.7204(2)	0.057(2)	0.062(2)	0.061(2)	0.001(1)	0.001(1)	-0.008(1)
C(4)	4a	0.0830(4)	0.1752(3)	0.6303(2)	0.047(2)	0.054(2)	0.053(2)	0.002(2)	0.002(2)	0.000(2)
C(5)	4a	0.1054(3)	0.0634(3)	0.6206(3)	0.041(2)	0.050(2)	0.060(2)	-0.003(2)	0.003(2)	0.004(2)
O(5)	4a	-0.1685(3)	0.2182(4)	0.6841(3)	0.062(2)	0.216(5)	0.091(2)	0.027(3)	0.007(2)	-0.008(3)
O(6)	4a	-0.0199(2)	0.0078(2)	0.6190(2)	0.056(1)	0.061(2)	0.053(1)	-0.013(1)	0.007(1)	-0.000(1)
C(6)	4a	0.1761(4)	0.0556(3)	0.5299(3)	0.048(2)	0.057(2)	0.060(2)	0.005(2)	0.009(2)	0.002(2)
O(7)	4a	-0.0119(4)	-0.0162(3)	0.7674(2)	0.107(3)	0.132(3)	0.056(2)	-0.046(2)	-0.003(2)	0.013(2)
C(7)	4a	0.2987(5)	-0.0580(4)	0.4305(3)	0.084(3)	0.077(3)	0.103(4)	0.003(3)	0.041(3)	-0.014(3)
C(8)	4a	0.1826(5)	-0.1159(4)	0.3911(3)	0.073(3)	0.098(4)	0.065(3)	0.023(3)	0.020(3)	-0.021(3)
C(9)	4a	0.1122(8)	-0.0784(7)	0.3185(5)	0.142(6)	0.180(7)	0.085(4)	0.080(6)	0.032(4)	-0.004(5)
C(10)	4a	0.011(1)	-0.132(1)	0.280(1)	0.14(1)	0.43(3)	0.121(8)	0.15(2)	-0.046(9)	-0.12(2)
C(11)	4a	-0.023(2)	-0.221(1)	0.312(1)	0.084(6)	0.39(3)	0.26(2)	0.02(1)	0.000(9)	-0.24(2)
C(12)	4a	0.0387(8)	-0.2594(7)	0.3850(7)	0.110(6)	0.165(7)	0.203(9)	-0.029(6)	0.053(6)	-0.108(8)
C(13)	4a	0.1431(6)	-0.2057(4)	0.4243(4)	0.098(4)	0.091(4)	0.106(4)	-0.009(3)	0.029(3)	-0.027(4)
C(14)	4a	0.3076(5)	0.3805(3)	0.5767(3)	0.064(3)	0.062(3)	0.058(2)	-0.008(2)	0.012(2)	0.003(2)
C(15)	4a	0.2690(5)	0.4871(3)	0.5602(3)	0.089(3)	0.055(3)	0.095(3)	-0.008(2)	0.018(3)	0.005(2)
C(16)	4a	-0.0802(4)	0.2240(4)	0.7390(3)	0.056(3)	0.083(3)	0.071(3)	0.008(2)	0.012(2)	0.001(2)
C(17)	4a	-0.0982(5)	0.2493(4)	0.8363(3)	0.092(4)	0.090(3)	0.077(3)	0.001(3)	0.031(3)	-0.007(3)
C(18)	4a	-0.0683(4)	-0.0250(3)	0.6975(3)	0.070(3)	0.066(3)	0.061(2)	-0.014(2)	0.006(2)	0.001(2)
C(19)	4a	-0.2045(5)	-0.0737(4)	0.6851(3)	0.082(3)	0.105(4)	0.079(3)	-0.034(3)	0.009(3)	0.009(3)

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