Crystal structure of (3a*S*,4*R*,5*S*,6*R*,6a*S*)-4,5,6-tris-benzoyloxy-1-methyl-hexahydro-cyclopent(*c*)isoxazole, $C_{28}H_{25}NO_7$

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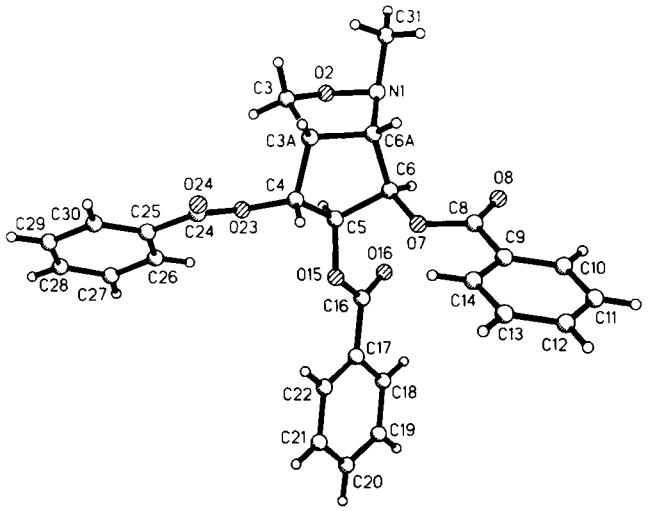

Source of material: The title isoxazolidine has been obtained by intramolecular nitrone cycloaddition (see ref. 1). Starting from methyl 6-iodo-2,3,4-tri-*O*-benzoyl-α-*D*-manno-pyranoside (see ref. 2), reaction with zinc and vitamin B12 as a catalyst afforded the corresponding 5-hexenose, which was transformed into the title compound without further purification by addition of *N*-methyl-hydroxylamine hydrochloride and pyridine as a base. Purification by flash chromatography on silica and crystallization from ethyl acetate/heptane yielded 39 % of the bicycle as a single diastereomer. The title compound is a precursor in a series of new glycosidase inhibitors (see ref. 3).

$C_{28}H_{25}NO_7$, orthorhombic, $P2_12_12_1$ (No. 19), a =5.823(1) Å, b =18.638(6) Å, c =23.147(5) Å, V =2512.1 Å^3, Z =4, $R(F)$ =0.087, $R_w(F^2)$ =0.120.

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

Crystal:	colourless needles, size 0.1 x 0.2 x 1.1 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.93 cm^{-1}
Diffractometer:	Nicolet P3
Scan mode:	Wyckoff
$T_{measurement}$:	293 K
$2\theta_{max}$:	48°
N(hkl)$_{unique}$:	2074
Criterion for I_o:	$I_o > 2\ \sigma(I_o)$
N($param$)$_{refined}$:	326
Program:	SHELXL-93

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

Atom	Site	x	y	z	U_{iso}
H(3A)	4*a*	0.735(3)	1.0135(6)	0.6677(4)	0.138
H(3B)	4*a*	0.614(3)	1.0889(6)	0.6714(4)	0.138
H(3A1)	4*a*	0.414(2)	1.0414(5)	0.7429(3)	0.097
H(4)	4*a*	0.491(2)	0.9355(5)	0.7884(3)	0.076
H(5)	4*a*	0.967(2)	0.9558(4)	0.7712(3)	0.072
H(6)	4*a*	0.979(2)	1.0417(4)	0.8425(3)	0.087
H(6A)	4*a*	0.584(2)	1.1040(5)	0.8148(3)	0.092
H(10)	4*a*	0.776(2)	1.0908(4)	1.0388(3)	0.077
H(11)	4*a*	0.534(2)	1.0718(5)	1.1158(4)	0.099
H(12)	4*a*	0.207(2)	1.0046(5)	1.1040(4)	0.096
H(13)	4*a*	0.120(2)	0.9571(5)	1.0159(4)	0.102
H(14)	4*a*	0.359(2)	0.9735(4)	0.9380(4)	0.079
H(18)	4*a*	1.367(2)	0.8233(6)	0.9215(4)	0.101
H(19)	4*a*	1.403(2)	0.7132(7)	0.9658(4)	0.115
H(20)	4*a*	1.120(3)	0.6275(7)	0.9554(5)	0.121
H(21)	4*a*	0.794(2)	0.6552(6)	0.9033(5)	0.117
H(22)	4*a*	0.754(2)	0.7667(6)	0.8611(4)	0.102
H(26)	4*a*	0.850(2)	0.7965(5)	0.6828(4)	0.097
H(27)	4*a*	0.934(2)	0.7000(5)	0.6226(5)	0.128
H(28)	4*a*	0.692(2)	0.6701(6)	0.5503(5)	0.117
H(29)	4*a*	0.336(2)	0.7226(5)	0.5429(4)	0.107
H(30)	4*a*	0.248(2)	0.8171(5)	0.6051(3)	0.087
H(31A)	4*a*	0.863(3)	1.2204(6)	0.7269(4)	0.269
H(31B)	4*a*	0.698(3)	1.2155(6)	0.7803(4)	0.269
H(31C)	4*a*	0.625(3)	1.1818(6)	0.7210(4)	0.269

Table 3. Final atomic coordinates and displacement parameters (in Å^2)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	4*a*	0.858(2)	1.1236(5)	0.7631(4)	0.19(1)	0.081(6)	0.081(6)	–0.041(8)	0.000(8)	0.009(5)
O(2)	4*a*	0.914(2)	1.0854(5)	0.7121(4)	0.161(9)	0.117(7)	0.101(6)	–0.017(7)	0.063(7)	0.004(5)
C(3)	4*a*	0.704(3)	1.0541(6)	0.6928(4)	0.20(2)	0.079(8)	0.063(6)	–0.01(1)	0.01(1)	–0.012(6)
C(3A)	4*a*	0.578(2)	1.0303(5)	0.7456(3)	0.103(9)	0.085(7)	0.056(5)	0.009(7)	0.015(6)	–0.003(5)
C(4)	4*a*	0.618(2)	0.9522(5)	0.7641(3)	0.064(7)	0.081(7)	0.046(5)	0.004(6)	0.011(5)	–0.013(5)
C(5)	4*a*	0.837(2)	0.9536(4)	0.7980(3)	0.066(7)	0.059(6)	0.056(5)	–0.014(6)	0.011(5)	–0.005(5)
C(6)	4*a*	0.825(2)	1.0240(4)	0.8328(3)	0.087(8)	0.085(7)	0.047(5)	–0.030(6)	0.020(6)	–0.011(5)

Table 3. (Continued)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(6A)	4*a*	0.698(2)	1.0761(5)	0.7930(3)	0.115(9)	0.068(6)	0.047(5)	0.005(7)	0.016(6)	–0.012(5)
O(7)	4*a*	0.691(1)	1.0095(3)	0.8840(2)	0.088(5)	0.078(4)	0.047(3)	–0.033(4)	0.013(4)	–0.020(3)
C(8)	4*a*	0.750(2)	1.0480(4)	0.9308(4)	0.076(7)	0.045(5)	0.055(5)	0.003(6)	–0.008(6)	–0.010(5)
O(8)	4*a*	0.904(1)	1.0893(3)	0.9323(2)	0.097(6)	0.087(5)	0.066(4)	–0.031(5)	0.002(4)	–0.015(4)
C(9)	4*a*	0.592(2)	1.0342(4)	0.9805(3)	0.058(6)	0.042(5)	0.041(4)	0.000(5)	0.004(5)	–0.004(4)
C(10)	4*a*	0.643(2)	1.0635(4)	1.0340(3)	0.072(7)	0.067(6)	0.052(5)	0.006(6)	–0.011(6)	–0.011(5)
C(11)	4*a*	0.499(2)	1.0522(5)	1.0799(4)	0.12(1)	0.082(8)	0.049(6)	0.022(8)	–0.004(7)	0.001(6)
C(12)	4*a*	0.305(2)	1.0123(5)	1.0728(4)	0.094(9)	0.086(8)	0.059(6)	0.016(7)	0.022(7)	0.016(6)
C(13)	4*a*	0.254(2)	0.9839(5)	1.0203(4)	0.098(9)	0.082(7)	0.074(6)	–0.008(7)	0.004(7)	0.000(6)
C(14)	4*a*	0.395(2)	0.9937(4)	0.9737(4)	0.084(8)	0.063(6)	0.051(5)	–0.001(6)	0.001(6)	0.006(5)
O(15)	4*a*	0.851(1)	0.8887(3)	0.8311(2)	0.056(4)	0.069(4)	0.073(4)	–0.020(4)	0.001(4)	–0.006(3)
C(16)	4*a*	1.053(2)	0.8773(6)	0.8572(4)	0.078(8)	0.086(8)	0.049(6)	–0.034(7)	0.014(6)	–0.022(6)
O(16)	4*a*	1.208(1)	0.9186(4)	0.8558(2)	0.071(5)	0.107(5)	0.068(4)	–0.036(5)	–0.003(4)	0.001(4)
C(17)	4*a*	1.057(2)	0.8064(6)	0.8857(4)	0.041(6)	0.106(9)	0.058(6)	–0.008(6)	0.001(5)	–0.030(6)
C(18)	4*a*	1.250(2)	0.7896(6)	0.9177(4)	0.068(8)	0.074(8)	0.110(8)	–0.021(7)	0.008(7)	–0.007(7)
C(19)	4*a*	1.272(2)	0.7233(7)	0.9440(4)	0.086(9)	0.100(9)	0.101(8)	–0.003(9)	–0.021(7)	–0.018(8)
C(20)	4*a*	1.104(3)	0.6725(7)	0.9384(5)	0.12(1)	0.092(9)	0.092(8)	0.02(1)	–0.005(9)	0.004(7)
C(21)	4*a*	0.911(2)	0.6890(6)	0.9072(5)	0.10(1)	0.081(8)	0.115(9)	–0.011(8)	0.009(8)	0.009(7)
C(22)	4*a*	0.887(2)	0.7560(6)	0.8815(4)	0.064(7)	0.111(9)	0.079(7)	–0.011(8)	–0.006(7)	–0.007(6)
O(23)	4*a*	0.656(1)	0.9023(3)	0.7167(2)	0.059(4)	0.073(4)	0.062(3)	–0.014(4)	0.002(4)	–0.029(3)
C(24)	4*a*	0.473(2)	0.8734(4)	0.6911(3)	0.068(7)	0.057(6)	0.039(5)	–0.017(6)	0.007(5)	0.004(4)
O(24)	4*a*	0.279(1)	0.8912(4)	0.7022(3)	0.056(4)	0.105(5)	0.078(4)	0.000(4)	0.000(4)	–0.031(4)
C(25)	4*a*	0.536(2)	0.8149(4)	0.6498(3)	0.056(6)	0.043(5)	0.049(5)	0.001(5)	–0.006(5)	–0.001(4)
C(26)	4*a*	0.745(2)	0.7815(5)	0.6551(4)	0.060(7)	0.081(7)	0.103(8)	–0.019(6)	–0.021(6)	–0.030(6)
C(27)	4*a*	0.797(2)	0.7249(5)	0.6182(5)	0.082(9)	0.089(8)	0.15(1)	0.003(7)	–0.021(9)	–0.059(8)
C(28)	4*a*	0.650(2)	0.7058(6)	0.5762(5)	0.092(9)	0.087(8)	0.114(9)	–0.016(8)	0.020(9)	–0.038(7)
C(29)	4*a*	0.441(2)	0.7377(5)	0.5708(4)	0.11(1)	0.080(8)	0.080(7)	–0.016(8)	–0.018(8)	–0.025(6)
C(30)	4*a*	0.388(2)	0.7938(5)	0.6086(3)	0.077(8)	0.073(7)	0.067(6)	–0.004(6)	–0.024(6)	–0.014(5)
C(31)	4*a*	0.752(3)	1.1911(6)	0.7464(4)	0.37(3)	0.071(8)	0.099(8)	0.01(1)	0.07(1)	0.002(6)

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