Crystal structure of (3*R*,4*S*,1″*S*,2″*S*)-3-acetoxy-1-benzyl- 4-(1″-benzyloxy-2″,3″-isopropylidenedioxy-propyl)-azetidin-2-one, $C_{25}H_{29}NO_6$

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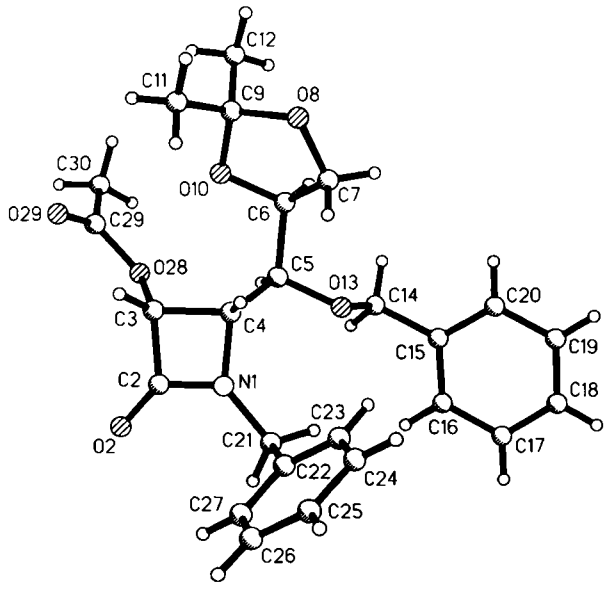

Source of material: The title compound (alternative name: 2-*O*-acetyl-3-amino-*N*,4-*O*-dibenzyl-3-deoxy-5,6-*O*-isopropylidene-L-idono-1,3-lactam; see ref. 1) was prepared by [2+2]-cycloaddition of (2*S*,3*S*)-2-*O*-benzyl-3,4-*O*-isopropylidenethreose *N*-benzylimine (see ref. 2) with acetoxyacetyl chloride in CH_2Cl_2/Et_3N at 243 K. The reaction gave a single diastereomer (> 95:5), which was crystallized from petroleum ether/ethyl acetate (65:35).

$C_{25}H_{29}NO_6$, orthorhombic, $P2_12_12$ (No. 18), a =13.541(3) Å, b =18.951(3) Å, c =9.378(2) Å, V =2406.5 Å^3, Z =4, $R(F)$ =0.079, $R_w(F^2)$ =0.213.

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

Crystal:	colorless block, size 0.4 x 0.75 x 0.75 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.86 cm^{-1}
Diffractometer:	Nicolet P3
Scan mode:	Wyckoff
$T_{measurement}$:	293 K
$2\theta_{max}$:	50°
$N(hkl)_{unique}$:	2282
Criterion for I_o:	$I_o > 2\,\sigma(I_o)$
$N(param)_{refined}$:	290
Programs:	SHELXS-86, SHELXL-93

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

Atom	Site	x	y	z	U_{iso}
H(3)	4*c*	0.2006(5)	0.1943(3)	0.3647(6)	0.066
H(4)	4*c*	0.3054(4)	0.1529(3)	0.1874(5)	0.055
H(5)	4*c*	0.2000(4)	0.2448(3)	–0.0012(6)	0.059
H(6)	4*c*	0.3382(5)	0.3120(3)	–0.0236(6)	0.066
H(7A)	4*c*	0.4503(5)	0.1983(5)	0.0800(9)	0.104
H(7B)	4*c*	0.4774(5)	0.2500(5)	–0.0461(9)	0.104
H(11A)	4*c*	0.3976(7)	0.3412(6)	0.433(1)	0.186
H(11B)	4*c*	0.5102(7)	0.3287(6)	0.403(1)	0.186
H(11C)	4*c*	0.4358(7)	0.2655(6)	0.394(1)	0.186
H(12A)	4*c*	0.3924(7)	0.4324(5)	0.248(1)	0.184
H(12B)	4*c*	0.4256(7)	0.4126(5)	0.093(1)	0.184
H(12C)	4*c*	0.5046(7)	0.4209(5)	0.214(1)	0.184
H(14A)	4*c*	0.2938(7)	0.2476(4)	–0.2435(6)	0.101
H(14B)	4*c*	0.1912(7)	0.2093(4)	–0.2307(6)	0.101
H(16)	4*c*	0.1681(6)	0.0940(4)	–0.3375(8)	0.098
H(17)	4*c*	0.2132(8)	0.0141(5)	–0.5129(9)	0.111
H(18)	4*c*	0.3608(8)	0.0261(5)	–0.6253(8)	0.110
H(19)	4*c*	0.4703(7)	0.1122(5)	–0.5567(9)	0.116
H(20)	4*c*	0.4231(7)	0.1932(4)	–0.3830(7)	0.098
H(21A)	4*c*	0.1834(6)	0.0665(3)	–0.0569(7)	0.086
H(21B)	4*c*	0.0969(6)	0.0368(3)	0.0365(7)	0.086
H(23)	4*c*	0.3494(7)	0.0238(4)	–0.0143(9)	0.108
H(24)	4*c*	0.4504(7)	–0.0645(5)	0.069(1)	0.129
H(25)	4*c*	0.394(1)	–0.1411(5)	0.238(1)	0.124
H(26)	4*c*	0.2369(9)	–0.1313(4)	0.3178(9)	0.111
H(27)	4*c*	0.1353(6)	–0.0471(3)	0.2286(7)	0.084
H(30A)	4*c*	0.1058(9)	0.3861(4)	0.194(1)	0.178
H(30B)	4*c*	0.1878(9)	0.4178(4)	0.294(1)	0.178
H(30C)	4*c*	0.0791(9)	0.4109(4)	0.349(1)	0.178

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)	4c	0.1657(4)	0.1191(2)	0.1266(5)	0.067(3)	0.050(2)	0.055(3)	−0.013(2)	−0.011(3)	0.003(2)
C(2)	4c	0.1035(5)	0.1431(4)	0.2247(8)	0.073(4)	0.064(4)	0.076(5)	−0.024(3)	0.008(4)	0.004(4)
O(2)	4c	0.0222(4)	0.1241(3)	0.2627(7)	0.092(4)	0.121(5)	0.123(5)	−0.046(4)	0.033(4)	−0.013(4)
C(3)	4c	0.1740(5)	0.2007(3)	0.2685(6)	0.064(3)	0.056(3)	0.046(3)	−0.008(3)	−0.008(3)	0.002(3)
C(4)	4c	0.2442(4)	0.1731(3)	0.1494(5)	0.052(3)	0.044(3)	0.040(3)	−0.004(2)	−0.007(2)	−0.002(2)
C(5)	4c	0.2614(4)	0.2201(3)	0.0227(6)	0.052(3)	0.053(3)	0.042(3)	0.004(3)	−0.005(2)	0.001(2)
C(6)	4c	0.3438(5)	0.2744(3)	0.0475(6)	0.065(4)	0.055(3)	0.046(3)	−0.004(3)	0.008(3)	0.003(3)
C(7)	4c	0.4482(5)	0.2470(5)	0.0482(9)	0.061(4)	0.101(5)	0.099(6)	−0.006(4)	0.017(4)	−0.017(5)
O(8)	4c	0.4974(5)	0.2912(5)	0.1443(8)	0.067(3)	0.186(7)	0.135(5)	0.012(4)	−0.011(4)	−0.053(6)
C(9)	4c	0.4285(5)	0.3297(4)	0.2275(8)	0.071(4)	0.077(4)	0.067(4)	−0.024(4)	−0.003(3)	0.006(4)
O(10)	4c	0.3343(3)	0.3040(2)	0.1864(5)	0.060(2)	0.067(2)	0.062(2)	−0.017(2)	0.003(2)	−0.010(2)
C(11)	4c	0.4444(7)	0.3150(6)	0.377(1)	0.128(8)	0.16(1)	0.080(6)	−0.071(8)	−0.027(5)	0.017(6)
C(12)	4c	0.4387(7)	0.4056(5)	0.193(1)	0.109(7)	0.098(6)	0.16(1)	−0.042(6)	−0.029(7)	0.036(7)
O(13)	4c	0.2894(3)	0.1760(2)	−0.0929(4)	0.072(3)	0.054(2)	0.040(2)	0.018(2)	−0.001(2)	−0.001(2)
C(14)	4c	0.2621(7)	0.2024(4)	−0.2273(6)	0.124(6)	0.080(4)	0.048(4)	0.039(5)	−0.008(4)	0.002(3)
C(15)	4c	0.2924(5)	0.1517(3)	−0.3396(6)	0.081(4)	0.065(3)	0.034(3)	0.013(3)	0.001(3)	0.004(3)
C(16)	4c	0.2294(6)	0.0982(4)	−0.3812(8)	0.075(4)	0.103(6)	0.067(4)	−0.005(4)	−0.002(4)	0.013(4)
C(17)	4c	0.2559(8)	0.0503(5)	−0.4873(9)	0.117(7)	0.090(5)	0.069(5)	−0.027(5)	−0.012(5)	0.000(4)
C(18)	4c	0.3439(8)	0.0572(5)	−0.5525(8)	0.133(8)	0.084(5)	0.057(4)	0.020(6)	−0.013(5)	−0.018(4)
C(19)	4c	0.4086(7)	0.1088(5)	−0.5136(9)	0.092(6)	0.130(7)	0.069(5)	0.005(6)	0.014(5)	−0.006(5)
C(20)	4c	0.3803(7)	0.1568(4)	−0.4075(7)	0.107(6)	0.094(5)	0.044(3)	−0.015(5)	−0.005(4)	−0.005(4)
C(21)	4c	0.1639(6)	0.0548(3)	0.0398(7)	0.089(5)	0.059(3)	0.067(4)	−0.017(4)	−0.018(4)	−0.006(3)
C(22)	4c	0.2313(5)	−0.0024(3)	0.0955(6)	0.073(4)	0.049(3)	0.054(3)	−0.010(3)	−0.009(3)	−0.005(3)
C(23)	4c	0.3249(7)	−0.0083(4)	0.0521(9)	0.106(6)	0.077(5)	0.087(5)	−0.018(5)	0.009(5)	0.002(4)
C(24)	4c	0.3861(7)	−0.0606(5)	0.103(1)	0.088(6)	0.091(6)	0.142(9)	0.003(5)	0.014(6)	−0.018(7)
C(25)	4c	0.353(1)	−0.1060(5)	0.202(1)	0.126(8)	0.068(5)	0.115(8)	0.017(5)	−0.034(7)	−0.015(5)
C(26)	4c	0.2603(9)	−0.1003(4)	0.2486(9)	0.154(9)	0.052(4)	0.071(5)	−0.013(5)	−0.026(6)	−0.004(4)
C(27)	4c	0.2000(6)	−0.0497(3)	0.1958(7)	0.093(5)	0.056(3)	0.060(4)	−0.016(3)	−0.007(4)	0.000(3)
O(28)	4c	0.1356(3)	0.2698(2)	0.2447(4)	0.063(2)	0.064(2)	0.053(2)	−0.003(2)	0.004(2)	−0.014(2)
C(29)	4c	0.1513(6)	0.3185(4)	0.345(1)	0.085(5)	0.084(5)	0.081(5)	−0.011(4)	0.027(5)	−0.029(5)
O(29)	4c	0.1810(6)	0.3059(4)	0.4596(6)	0.164(7)	0.114(5)	0.068(3)	−0.014(5)	0.001(4)	−0.036(3)
C(30)	4c	0.1291(9)	0.3895(4)	0.291(1)	0.153(9)	0.078(5)	0.124(8)	−0.002(6)	0.042(7)	−0.020(6)

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