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Note

Synthesis and X-ray structure of a C5–C4-linked glucofuranose–oxazolidin-2-one

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ABSTRACT

The formation of (4*R*)-4-carbamoyl-4-[(4*R*)-3-*O*-benzyl-1,2-*O*-isopropylidene-β-L-threofuranos-4-*C*-yl]-oxazolidin-2-one instead of expected imidazolidin-2,4-dione (hydantoin) derivative from 5-amino-5-cyano-5-deoxy-3-*O*-benzyl-1,2-*O*-isopropylidene-α-D-glucofuranose or 3-*O*-benzyl-1,2-*O*-isopropylidene-α-D-*xylo*-hexofuranos-5-ulose under Bucherer–Bergs reaction conditions is reported. Single crystal X-ray diffraction data revealed that $^{3}T_{4}$ is the prefered conformation for the furanose ring, while E_2 and $^{2}T_{1}$ conformations are adopted by the 1,3-dioxolane and 2-oxazolidinone five-membered rings, respectively.

Derivatives of oxazolidin-2-one represent an important class of synthetic biologically active agents.[1–4] Some of them have also been therapeutically applied (e.g., Zolmitriptan, used as a drug for acute treatment of migraine).[5] In addition, functionalized chiral oxazolidin-2-ones, known as Evans' chiral auxiliaries,[6,7] have widely been applied as versatile chiral building blocks in asymmetric syntheses of biologically active compounds or their intermediates.[8–12] Among them, a glycoconjugate derived from D-mannitol has also been introduced.[13] Owing to the above mentioned utilities and emerging new applications of oxazolidin-2-ones, considerable attention has been focused on their synthesis. Generally, they are prepared from 1,2-amino alcohols with phosgene (or diphosgene) and dialkyl carbonates (or alkyl chloroformates).[14–16] The construction of the oxazolidin-2-one ring, its application and modification have been surveyed in two recent reviews.[17,18]

Recently, we have described the Bucherer–Bergs reaction of some hexofuranos-5-uloses having unprotected 6-OH group, affording C5–C4-linked glucofuranose–oxazolidin-2-one instead of expected corresponding imidazolidin-2,4-dione (hydantoin) derivative.[19,20] As a continuation of this work, we have used 3-*O*-benzyl-1,2-*O*-isopropylidene-α-D-*xylo*-hexofuranos-5-ulose (**1**) as a starting 5-ulose (Scheme 1). In this case, a mixture of products resulted and, although the oxazolidin-2-one (as seen from the comparison of TLC and NMR data of the crude reaction mixture) represented the main product, its isolation and purification were problematic. Due to very similar R_f values of several co-products, supposing a possibility of both *R* and *S* diastereomers of corresponding hydantoin and amino nitrile, chromatographic separation of oxazolidin-2-one derivative of adequate purity, was unsuccessful. Therefore, we have decided on a two-step approach. Thus, starting from 5-ulose **1**,[21,22] the corresponding amino nitrile was prepared in the first step as a mixture of 5*R* and 5*S* isomers (in the ratio of 60:40 as determined by ^{1}H NMR spectroscopy). The pure 5*R* isomer – 5-amino-5-cyano-5-deoxy-3-*O*-benzyl-1,2-*O*-isopropylidene-α-D-glucofuranose [alternative name: (5*R*)-5-amino-5-cyano-5-deoxy-3-*O*-benzyl-1,2-*O*-isopropylidene-α-D-*xylo*-hexofuranose] (**2**) was obtained using column chromatography and subsequent several recrystallizations of the component that eluted first (R_f 0.72). Our efforts to obtain also the pure 5*S* isomer were unsuccessful. Even repeated column chromatography of the slower moving oily component (R_f 0.65) always afforded product contaminated with ca. 15% (as indicated by ^{1}H NMR spectroscopy) of the 5*R* isomer. In the second step, reaction of amino nitrile **2** with ammonium carbonate gave desired (4*R*)-4-carbamoyl-4-[(4*R*)-3-*O*-benzyl-1,2-*O*-isopropylidene-β-L-threofuranos-4-*C*-yl]-oxazolidin-2-one [alternative name: (4*R*,4*R*)-4-*C*-(4-carbamoyl-2-oxooxazolidin-4-yl)-3-*O*-benzyl-1,2-*O*-isopropylidene-β-L-threofuranose] (**3**) (Scheme 1).

The structure of **3** was established on the basis of NMR and mass spectral data. The *R* configuration at C4 position of oxazolidinone ring (this position coincides with the C5 position of the hexofuranose moiety) was confirmed by X-ray crystallography (Fig. 1, Table 1). The absolute configuration at stereocentres C1,

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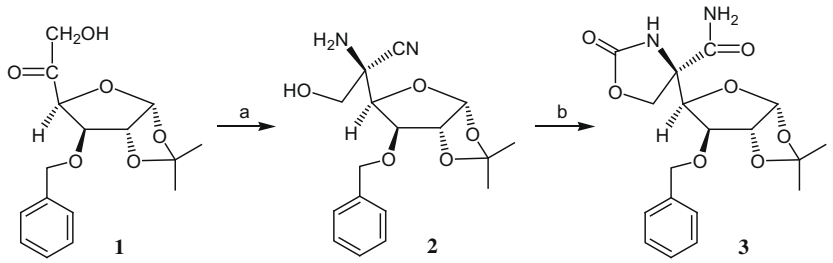

Scheme 1. Reagent and conditions: (a) NH_3 in MeOH, NaCN, NH_4Cl, 20 °C, 48 h; (b) $(NH_4)_2CO_3$, aq EtOH, 60 °C, 8 h.

C2, C3 and C4 of the furanose moiety in **3** was assigned on the basis of the known arrangement in analogous D-glucofuranose derivatives. Analogously, the *R* configuration at C5 position in amino nitrile **2** was then deduced from the known arrangement of atoms in **3** and considering the usual mechanism of the Bucherer–Bergs reaction[23,24] that reaction with ammonium carbonate should proceed via an attack of carbon dioxide at amino group of **3**, leaving the C5 position of hexofuranose unaffected.

The presence of a 1,3-dioxolane ring bonded to a furanose ring at the 1,2-position imposes some conformational rigidity on compound **3**. An inspection of the relevant torsion angle values (Table 2) and the calculated values of ring puckering parameters[25] $Q = 0.373(3)$ Å and $\Phi = 304.1(4)°$ revealed that O4–C1–C2–C3–C4 furanose ring adopts the $^3T_4$ ($^{C3}T_{C4}$) conformation with C3 atom lying in the *endo* and C4 *exo* direction with respect to the O4–C1–C2 reference plane. Analogously, the puckering parameters $Q = 0.174(3)$ Å, $\Phi = 256.9(9)°$ and the values of relevant dihedral angles (Table 2) are indicative of a conformation very close to E_2 (envelope on C2) for five-membered 1,3-dioxolane ring (O1–C1–C2–O2–C10). Considering the puckering parameters $Q = 0.271(3)$ Å, $\Phi = 56.4(6)°$ and the relevant torsion angle values (Table 2), the conformation of the five-membered oxazolidine ring (O6–C6–C5–N2–C8) can be described mainly as a $^2T_1$ ($^{C5}T_{C6}$, twisted on C6–C5).

Inspection of the molecular packing in the unit cell revealed 3 principal hydrogen bonds (see Fig. 2, Table 3) and 4 weak contacts, among them 3 were intramolecular ([d], [f], [g]) and 4 were intermolecular ([a], [b], [c], [e]). The first level hydrogen bond descriptors based on the graph-set theory[26] comprise: chains C(8) for [a], C(7) for [b], C(4) for [c] and C(6) for [e], strings (intramolecular contacts) S(5) for [d], [f] and [g]. The second level includes: ring R1,2(7) for [b,c] and chains C2,2(15) for [a,b], C2,2,(11) for [a,b], [a,e], [b,c] and [c,e], C2,2(14) for [a,c] and [a,e], C2,2(13) for [b,e], C2,2(10) also for [b,e] and C2,2(9) for [c,e]. Assignment of the hydrogen bond descriptors, based on the graph-set theory[26] was performed using the program PLUTO.[27]

Table 1
Crystallographic and experimental data for compound **3**[a]

Empirical formula	$C_{18}H_{22}N_2O_7$
Formula weight	378.38
Temperature, *T* (K)	183(2)
Wavelength, λ (Å)	0.71073
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
a (Å)	6.8333(1)
b (Å)	8.7368(1)
c (Å)	30.6410(2)
Unit-cell volume *V* (Å^3)	1829.30(4)
Formula per unit cell, *Z*	4
D_{calcd} (g/cm^3)	1.374
Absorption coefficient, μ (mm^{-1})	0.107
F(0 0 0)	800
Crystal size (mm)	0.52 × 0.10 × 0.02
θ Range for data collection (°)	2.42–27.49
Index ranges	$-8 \le h \ge 8$ $-11 \le k \ge 11$ $-39 \le l \ge 39$
Reflections collected	23905
Independent reflections (R_{int})	2434 (0.0609)
Completeness to θ (%)	99.9
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	2434/42/310
Goodness-of-fit on F^2	1.014
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0446$, $wR_2 = 0.1047$
R indices (all data)	$R_1 = 0.0576$, $wR_2 = 0.1123$
Largest difference peak and hole (e/Å^{-3})	0.398 and −0.295

[a] Standard deviations in parentheses.

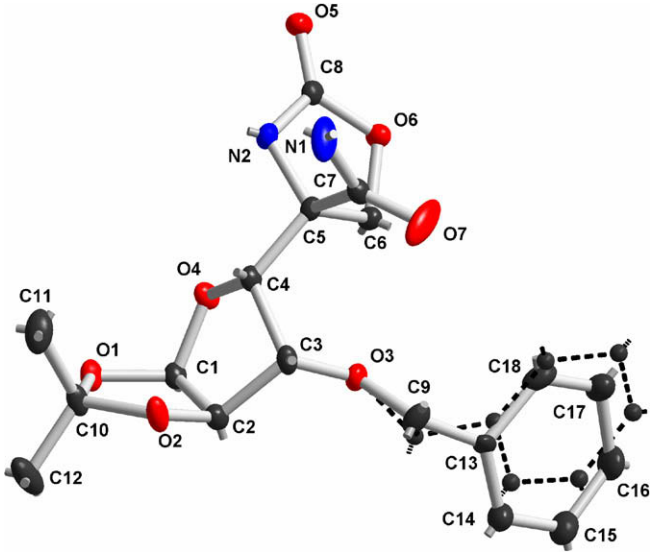

Figure 1. A perspective drawing of **3** showing the atom-numbering. Hydrogen atoms are masked by short bonds for clarity. Displacement ellipsoids are shown at 30% probability level. The structure is disordered in the orientation of the phenyl ring. The occupancy was refined to 0.53(2) and 0.47(2), respectively; the part with lower occupancy is shown in broken lines. The dihedral angle between the two disordered phenyl rings is 18.6(9)°.

1. Experimental

1.1. General methods

The ^1H and ^{13}C NMR spectra (in $CDCl_3$, internal standard Me_4Si) were recorded on a Bruker Avance DPX 300 instrument (equipped with gradient-enhanced spectroscopy kit GRASP for generation of *Z* gradient up to 50 Gauss/cm) operating at 300.13 and 75.46 MHz, respectively. The quaternary carbon atoms were identified on the basis of a semiselective INEPT experiment and a 1D INADEQUATE pulse sequence technique. When reporting assignments of ^{13}C NMR signals, data for the phenyls are identified by a prime. The EI and CI (using pyridine as a reactive agent) mass spectra

Table 2
Relevant torsion angles (°) for five-membered rings in compound **3**[a]

Furanose		1,3-Dioxolane		Oxazolidin-2-one	
O4–C1–C2–C3	−12.4(3)	O1–C1–C2–O2	−16.9(3)	O6–C6–C5–N2	−26.7(2)
C1–C2–C3–C4	30.4(3)	C1–C2–O2–C10	18.8(3)	C6–C5–N2–C8	−23.9(3)
C2–C3–C4–O4	−38.7(3)	C2–O2–C10–O1	−13.2(3)	C5–N2–C8–O6	11.3(3)
C3–C4–O4–C1	32.7(3)	O2–C10–O1–C1	1.5(3)	N2–C8–O6–C6	8.1(3)
C4–O4–C1–C2	−12.4(3)	C10–O1–C1–C2	9.6(3)	C8–O6–C6–C5	−22.8(3)

[a] Standard deviations in parentheses.

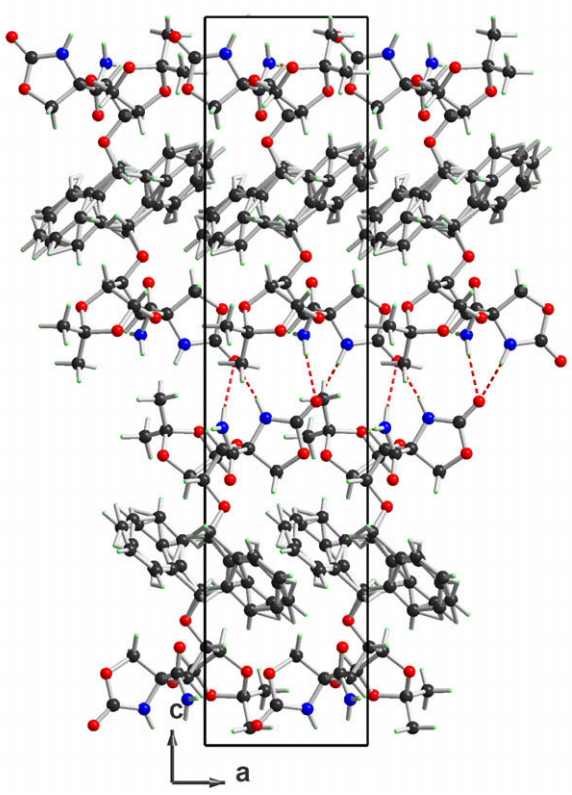

Figure 2. Unit cell contents of **3** in projection along the *b*-axis. Hydrogen bonds [a], [b] and [c] (see Table 3 for details) are shown as broken lines.

Table 3
Hydrogen bonds (Å) and angles (°) for **3**[a]

Notation	D–H···A	D–H	H···A	D···A	D–H···A
a	N1–H1A···O1^{i}	0.88	2.04	2.910(3)	172
b	N1–H1B···O5^{ii}	0.88	2.25	3.128(3)	174
c	N2–H2···O5^{ii}	0.88	2.13	2.840(3)	138
d	N1–H1B···N2	0.88	2.42	2.763(4)	103
e	C3–H3···O6^{iii}	1.00	2.40	3.260(3)	143
f	C6–H6A···O7	0.99	2.43	2.852(4)	105
g	C6–H6B···O4	0.99	2.55	2.966(3)	105

[a] Standard deviations in parentheses. *Symmetry codes*: (i) *x*, *y* − 1, *z*; (ii) *x* + 1/2, −*y* + 1/2, −*z*; (iii) *x* + 1, *y*, *z*.

(70 eV) were obtained on a Finnigan MAT SSQ 710 instrument. Specific rotations were determined on a Perkin–Elmer 241 polarimeter (10 cm cell). Microanalyses were performed on a Fisons EA 1108 analyzer. Melting points were determined with a Boetius PHMK 05 microscope. Column chromatography was performed as flash chromatography on Silica Gel 60 (E. Merck, 0.063–0.200 mm).

1.2. 5-Amino-5-cyano-5-deoxy-3-*O*-benzyl-1,2-*O*-isopropylidene-α-D-glucofuranose [alternative name: (5*R*)-5-amino-5-cyano-5-deoxy-3-*O*-benzyl-1,2-*O*-isopropylidene-α-D-*xylo*-hexofuranose] (2)

To a soln of 5-ulose **1** (3.08 g, 10 mmol) in MeOH (150 mL) were added NaCN (0.98 g, 20 mmol) and NH_4Cl (1.07 g, 20 mmol). The mixture was saturated with ammonia and stirred for 48 h at room temperature. Methanol was then evaporated under reduced pressure, the residue was dissolved in water (60 mL) and the product extracted with diethyl ether (3 × 30 mL). The combined extracts were washed with satd aq soln of NH_4HCO_3 (20 mL) and water (20 mL), successively, dried over $MgSO_4$, filtered and concentrated under diminished pressure. The crude product (1.74 g, 52%) was chromatographed on a column of silica gel using 2:1 EtOAc–hexane as an eluent. The fractions having R_f 0.72 were collected and evaporated to give almost pure product. Three recrystallizations from MeOH afforded white crystals of **2** (1.04 g, 31%) with mp 131–132 °C; $[\alpha]_D$ −44 (*c* 1, MeOH); ^{1}H NMR ($CDCl_3$): δ 7.45–7.31 (m, 5H, aromatic), 6.05 (d, 1H, $J_{1,2}$ = 3.7 Hz, H-1), 4.86 and 4.50 (2d of ABq, each 1H, $J_{Ha,Hb}$ = 12.1 Hz, CH_2Ph), 4.68 (d, 1H, H-2), 4.14 (d, 1H, $J_{3,4}$ = 3.8 Hz, H-4), 4.01 (d, 1H, H-3), 4.01 and 3.59 (2d of ABq, each 1H, $J_{Ha,Hb}$ = 11.1 Hz, H-6), 2.19 (br s, 2H, NH_2), 1.47 and 1.33 (2s, each 3H, Me_2C); ^{13}C NMR ($CDCl_3$): δ 136.3 (C-1′), 128.8 (C-2′ and C-6′), 128.4 (C-4′), 128.0 (C-3′ and C-5′), 121.3 (CN), 112.2 (CMe_2), 105.5 (C-1), 81.3 (C-2), 81.2 (C-4), 80.8 (C-3), 71.7 (CH_2Ph), 67.2 (C-6), 56.0 (C-5), 26.8 and 26.2 [$(CH_3)_2C$]. Anal. Calcd for $C_{17}H_{22}N_2O_5$: C, 61.10; H, 6.63; N, 8.38. Found: C, 61.27; H, 6.72, N, 8.30.

Evaporation of the fractions with R_f 0.65 (following the elution of **2**) gave, even after three repeated separations, corresponding 5*S* isomer of **2** (0.7 g, 21%) in only ca. 85% purity as an oil with $[\alpha]_D$ −32 (*c* 1, MeOH); ^{1}H NMR ($CDCl_3$): δ 7.43–7.28 (m, 5H, aromatic), 6.02 (d, 1H, $J_{1,2}$ = 3.7 Hz, H-1), 4.71 and 4.62 (2d of ABq, each 1H, $J_{Ha,Hb}$ = 11.0 Hz, CH_2Ph), 4.64 (d, 1H, H-2), 4.29 (d, 1H, $J_{3,4}$ = 3.7 Hz, H-4), 4.25 (d, 1H, H-3), 3.69 (s, 2H, H-6), 1.49 and 1.33 (2s, each 3H, Me_2C); ^{13}C NMR ($CDCl_3$): δ 136.3 (C-1′), 128.5 (C-2′ and C-6′), 128.2 (C-4′), 128.0 (C-3′ and C-5′), 120.8 (CN), 112.2 (CMe_2), 105.2 (C-1), 83.3 (C-2), 81.7 (C-4), 79.4 (C-3), 72.7 (CH_2Ph), 65.3 (C-6), 55.9 (C-5), 26.7 and 26.1 [$(CH_3)_2C$]. Anal. Calcd for $C_{17}H_{22}N_2O_5$: C, 61.10; H, 6.63; N, 8.38. Found: C, 61.27; H, 6.71, N, 8.46.

1.3. (4*R*)-4-Carbamoyl-4-[(4*R*)-3-*O*-benzyl-1,2-*O*-isopropylidene-β-L-threofuranos-4-*C*-yl]-oxazolidin-2-one [alternative name: (4*R*,4*R*)-4-*C*-(4-carbamoyl-2-oxooxazolidin-4-yl)-3-*O*-benzyl-1,2-*O*-isopropylidene-β-L-threofuranose] (3)

A mixture of amino nitrile **2** (668 mg, 2 mmol) and $(NH_4)_2CO_3$ (864 mg, 9 mmol) in 50% aq EtOH (10 mL) was stirred for 8 h at 60 °C. Ethanol was then evaporated and the product extracted with $CHCl_3$. After complete solvent removal under diminished pressure, the residue was chromatographed on a column of silica gel using 10:1 $CHCl_3$–MeOH as an eluent. The fractions having R_f 0.45 were collected and evaporated to give **3** (582 mg, 77%). Recrystallization

from MeOH afforded white needles with mp 198–199 °C; $[\alpha]_D$ +48 (*c* 1, MeOH); ^{1}H NMR ($CDCl_3$): δ 7.42–7.31 (m, 5H, aromatic), 6.63 and 5.55 (2br s, each 1H, NH_2), 5.69 (br s, 1H, NH), 5.97 (d, 1H, $J_{1,2}$ = 3.4 Hz, H-1), 4.69 (d, 1H, $J_{3,4}$ = 3.2 Hz, H-4), 4.58 (d, 1H, H-2), 4.62 and 4.49 (2d of ABq, each 1H, $J_{Ha,Hb}$ = 11.6 Hz, H-5), 4.38 and 4.32 (2d of ABq, each 1H, $J_{Ha,Hb}$ = 12.1 Hz, CH_2Ph), 4.14 (d, 1H, H-3), 1.53 and 1.32 (2s, each 3H, Me_2C); ^{13}C NMR ($CDCl_3$): δ 172.8 (*C*ONH_2), 159.1 (O*C*ONH), 135.9 (C-1′), 128.9 (C-2′ and C-6′), 128.7 (C-3′ and C-5′), 128.6 (C-4′), 112.6 (*C*Me_2), 105.1 (C-1), 81.6 (C-3), 80.9 (C-4), 80.8 (C-2), 72.1 (C-5), 70.8 (CH_2Ph), 63.1 (C-4 of oxazolidinone), 26.9 and 26.3 [(CH_3)$_2$C]; EIMS (70 eV): *m*/*z* 334 (44%, [M−$CONH_2$]$^+$), 249, 129, 92, 91 (100%), 65, 43. CIMS: *m*/*z* 458 (M+C_5H_5NH)$^+$. Anal. Calcd for $C_{18}H_{22}N_2O_7$: C, 57.10; H, 5.86; N, 7.40. Found: C, 57.33; H, 5.94, N, 7.31.

1.4. X-ray techniques

Single crystals of adequate quality, suitable for X-ray diffraction, were obtained by slow crystallization of **3** from MeOH under cooling in refrigerator.

Crystallographic data were collected on a Siemens SMART CCD diffractometer. Preliminary orientation matrix was obtained from the first frames using Siemens SMART software.[28] Final cell parameters were obtained by refinement of 8192 reflections using Siemens SAINT software.[28] The data were empirically corrected for absorption and other effects using SADABS program[29] based on the method of Blessing.[30] Crystal and experimental data are given in Table 1.

The structure was solved by direct methods and refined by full-matrix least-squares on all F^2 data using Bruker SHELXTL.[31] The non-H atoms were refined anisotropically. All the hydrogen atoms were constrained to the ideal geometry using an appropriate riding model. Molecular graphics were obtained using the program DIAMOND.[32]

2. Supplementary data

Complete crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 728464. Copies of this information may be obtained free of charge from the Director, Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: + 44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or via: www.ccdc.cam.ac.uk).

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