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Note

Synthesis of 2-alkyl(aryl)amino-(3,4,6-tri-*O*-acetyl-1,2-dideoxy-α-D-glucopyrano)[2,1-*d*]-2-oxazolines and ethyl 2-[3-alkyl(aryl)ureido]-2-deoxy-β-D-glucopyranosides from acetylated 2-[3-alkyl(aryl)ureido]-2-deoxy-D-glucopyranoses

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Carbohydrate oxazolines, which are valuable intermediates in the synthesis of aminodisaccharides[1,2] and glycosides[2–5], have been prepared by cyclisation of 1,3,4,6-tetra-*O*-acyl-2-acylamino-2-deoxy-α- or -β-glucopyranose promoted by hydrogen bromide–acetic acid[6] or Lewis acid catalysts[2,4,5,7,8] and by cyclisation of 1-propenyl β-glycosides[9].

We now report a new type of sugar oxazoline, namely, 2-alkyl(aryl)amino-(3,4,6-tri-*O*-acetyl-1,2-dideoxy-α-D-glucopyrano)[2,1-*d*]-2-oxazolines by cyclisation of acetylated 2-[3-alkyl(aryl)ureido]-2-deoxy-D-glucopyranoses[10–16].

The synthesis of 2-alkyl(aryl)amino-(1,2-dideoxy-α-D-glucopyrano)[2,1-*d*]-2-thiazoline hydrobromides from 1,3,4,6-tetra-*O*-acetyl-2-[3-alkyl(aryl)thioureido]-2-deoxy-D-glucopyranoses by treatment with hydrogen bromide–acetic acid has been described[17]. Similarly, sugar oxazolines could be obtained from ureido derivatives. When 1,3,4,6-tetra-*O*-acetyl-2-deoxy-2-(3-phenylureido)-α-D-glucopyranose (**1**) and 1,3,4,6-tetra-*O*-acetyl-2-deoxy-2-(3-methylureido)-α-D-glucopyranose (**3**) were treated with (HBr–AcOH), the corresponding 2-alkyl(aryl)amino-(1,2-dideoxy-α-D-glucopyrano)[2,1-*d*]-2-oxazoline hydrobromides (**5**) could not be isolated, but further reaction with ethanol gave the ethyl β-glucosides **6** and **7** (from **1**) and **8** (from **3**). Acetylation of **6** and **8** gave the triacetates **7** and **9**. Zemplén deacetylation of **7** gave **6**.

The structures **6–9** were indicated by elemental analyses (see Experimental) and ^{1}H- and ^{13}C-n.m.r. data (Tables I–III). The *O*-glycosylic linkage is indicated by the low value of the chemical shift of the resonance of H-1 (~4.6–4.4 p.p.m.).

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TABLE I

^{1}H-N.M.R. CHEMICAL SHIFT DATA[a] (δ, P.P.M.)

Compound	*H-1*	*H-2*	*H-3*	*H-4*	*H-5*	*H-6*	*H-6'*	*NH*	*OEt*		NCH_3	*Aromatic*	*OAc*
									CH_2	CH_3			
7[b]	4.60 d	3.72 m	5.30 t	5.08 t	3.72 m	4.29 dd	4.11 dd	7.71 s	3.72 m	1.27 t		7.39–6.96 m	2.06 s (3 H) 2.05 s (3 H) 2.03 s (3 H)
9[c]	4.62 d	3.68 m	5.26 t	5.02 t	3.68 m	4.28 dd	4.09 dd	5.53 m	3.68 m	1.19 t	2.71 d		2.03 s (3 H) 2.01 s (3 H) 1.97 s (3 H)
11[b]	6.00 d	4.25 m	5.33 dd	4.92 m	3.91 m	4.19 m		7.40 s				7.41–7.00 m	2.11 s (3 H) 2.09 s (3 H) 1.93 s (3 H)
12[b]	6.01 d	4.24 m	5.30 t	4.95 ddd	3.90 m	4.22 m						7.32 dd	2.12 s (3 H) 2.09 s (3 H) 1.98 s (3 H)
13[c]	5.91 d	4.17 m	5.24 m	4.90 m	3.80 m	4.17 m		7.35 m			2.86		2.08 s (3 H) 2.06 s (3 H) 2.04 s (3 H)
14[b,d]	5.85 d	4.05 m	5.10 t	4.96 td	4.05 m	4.20 dd	4.05 m	6.25 s				7.70–7.00 m	2.13 s (3 H) 2.07 s (3 H) 1.92 s (3 H)
15[b]	4.32 d	3.91 m	5.22 t	5.07 t	3.65 m	4.29 dd	4.10 dd	7.85 s 6.04 d	3.91 m	1.22 t		7.21 dd	2.05 s (3 H)

[a]In $CDCl_3$. [b]At 200 MHz. [c]At 90 MHz. [d]Ref. 16.

TABLE II

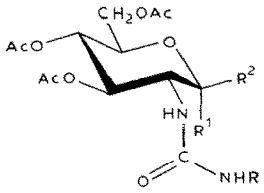
^{1}H-N.M.R. J VALUES[a]

Compound	$J_{1,2}$	$J_{2,3}$	$J_{2,4}$	$J_{3,4}$	$J_{4,5}$	$J_{5,6}$	$J_{5,6'}$	$J_{6,6'}$	$J_{2,NH}$
7	8.4	9.3		9.3	9.3	7.3	2.2	−12.2	8.7
9	8.0	10.0		10.0	10.0	6.0	2.0	−13.0	
11	6.8	2.4	1.6	2.5	9.1	4.9	4.3		
12	6.8	2.6	1.1	2.5	9.1	4.6	4.3		
13	6.5	3.0	0.3	2.3	9.1	4.6	4.2		
14	7.5	2.8	0.9	2.7	9.1	6.5	2.4	−12.1	
15	8.3	9.6		9.6	9.6	4.9		−12.3	5.6

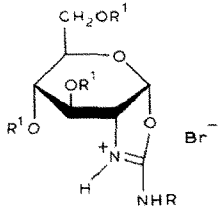
[a]Conditions described in Table I.

Assignment of their β-D configuration is based on the low $[\alpha]_D$ values similar to that[18] for the *O*-methyl analogue of **7** and the large value (~8 Hz) of $J_{1,2}$.

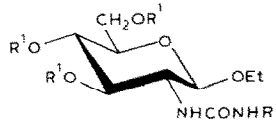
The amino-oxazolines are suitable starting materials for the synthesis of glycosides, and the tin chloride procedure described by Srivastava[8] is especially attractive. This reagent induced intramolecular cyclisation of 2-acetamido-1,3,4,6-tetra-*O*-acetyl-2-deoxy-α- and -β-D-glucopyranose to produce **10** in high yield. Likewise, treatment of **1–3** with anhydrous tin chloride gave **11–13** in yields of >80%, and **11** was also obtained from **4**. Compounds **11–13** had i.r. absorptions for NH (3360–3260 cm^{-1}) and C=N (1690–1665 cm^{-1}) of oxazoline ring[5,9,19]. The ^{13}C-



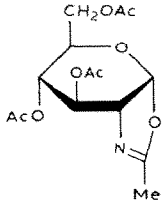
1 R = Ph, R^1 = OAc, R^2 = H
2 R = 4-ClC$_6$H$_4$, R^1 = OAc, R^2 = H
3 R = Me, R^1 = OAc, R^2 = H
4 R = Ph, R^1 = H, R^2 = OAc



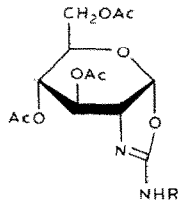
5 R^1 = H or Ac



6 R = Ph, R^1 = H
7 R = Ph, R^1 = Ac
8 R = Me, R^1 = H
9 R = Me, R^1 = Ac
15 R = 4-ClC$_6$H$_4$, R^1 = Ac



10



11 R = Ph
12 R = 4-ClC$_6$H$_4$
13 R = Me

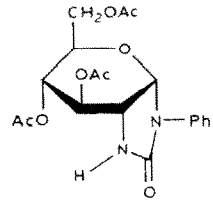


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TABLE III

^{13}C-N.M.R. CHEMICAL SHIFTS[a]

Compound	*C-1'*	*C-2'*	*C-3'*	*C-4'*	*C-5'*	*C-6'*	*NCN* ‖ *O*	*NCO* ‖ *N*	*Acetate*		*Aromatics*				*Ethyl*		*Methyl*
									CO	CH_3	*C-1*	*C-2,6*	*C-3,5*	*C-4*	CH_2	CH_3	
6[b]	101.2	56.4	75.0	71.0	77.0	61.3	155.5				140.7	117.8	128.8	121.2	63.8	15.3	
7[c]	101.5	58.2	72.6	68.7	71.5	62.2	155.9		170.5 169.4 167.6	20.6 20.5 20.4	138.7	120.3	128.8	121.9	65.8	14.9	
8[b]	101.2	56.8	75.6	73.2	76.9	61.2	159.3								63.7	15.3	26.6
11[c]	98.7	53.1	71.1	68.9	68.2	63.2		157.4	170.4 169.4 169.1	20.6	138.5	119.1	129.4	123.3			
12[c]	98.9	62.7	70.8	68.2	67.7	63.2		157.1	170.6 169.5 169.4	20.8 20.6 20.5	137.4	120.4	128.9	127.9			
13[c,d]	99.1	63.1	71.0	68.2	67.0	63.1		161.5	170.4 169.4 169.1	20.6 20.5 20.4							28.7
14[c]	83.2	50.1	70.3	67.8	67.1	63.1	158.1		170.3 169.6	20.6 20.5 20.3	137.1	121.2	128.9	124.9			
15[c,d]	101.8	55.3	72.7	68.8	71.5	62.3	156.0		170.7 170.6 169.5	20.7 20.6 20.6	137.4	121.7	128.8	128.0	66.1	15.0	

[a]At 50.3 MHz. [b]In $(CD_3)_2SO$. [c]In $CDCl_3$. [d]Assignments confirmed by heteronuclear double resonance.

n.m.r. signals for C-1′ (~99 p.p.m.) and C-2′ (~63 p.p.m.) reflected greater deshielding (~ −8 and −11 p.p.m., respectively) than in the corresponding ureas[16]. These shifts are similar to those described for 2-oxazolines[8] and rule out the isomeric glucopyrano[2,1-*d*]imidazolidine-2-one structures (**14**) [δ ~83 (C-1′) and ~50 (C-2′)]. The ^{13}C-n.m.r. data for **14**[16] are included in Table III for purposes of comparison.

The $^{3}J_{H,H}$ values for **11–13** indicate a marked distortion of the $^{4}C_{1}$(D) conformation. Thus, the derived[20,21] values of $\phi_{1,2}$ (20–60°), $\phi_{2,3}$ (~120°), $\phi_{3,4}$ (~120°), and $\phi_{4,5}$ (173°) suggest the equilibrium $B_{2,5} \rightleftharpoons {}^{o}S_{2} \rightleftharpoons {}^{o}H_{5} \rightleftharpoons {}^{4}C_{1}$.

The glycosylating ability of the 2-amino[2,1-*d*]-2-oxazolines **11–13** was illustrated by their reactions with boiling ethanol, which gave >50% of the ethyl β-glucosides **7, 9,** and **15**, respectively. The use of **11–13** in the synthesis of 2-alkyl-(aryl)ureido-disaccharides is under investigation.

EXPERIMENTAL

General methods. — Solutions were concentrated *in vacuo* at <50°. Melting points were determined with a Gallenkamp apparatus and are uncorrected. Optical rotations were measured at 20 ±5° with a Perkin–Elmer 141 polarimeter (10-cm pathlength). I.r. spectra (KBr disc) were recorded with a Perkin–Elmer 399 spectrophotometer, and u.v. spectra with a Pye–Unicam SP8-250 instrument. T.l.c. was conducted on Silica Gel GF_{254} (Merck) with benzene–ether (3:2), using detection with u.v. light or iodine vapor.

The ^{1}H- (200 MHz) and ^{13}C-n.m.r. spectra (50 MHz) were determined mainly with Varian XL-200 and Bruker AC 200-E spectrometers. Assignments were confirmed as appropriate by double resonance, APT, and DEPT experiments.

*Ethyl 2-deoxy-2-(3-phenylureido)-β-*D*-glucopyranoside* (**6**) *and its triacetate* (**7**). — (*a*) 1,3,4,6-Tetra-*O*-acetyl-2-deoxy-2-(3-phenylureido)-α-D-glucopyranose[11,16] (**1**: 1.0 g, 2.2 mmol) was added to a saturated solution of hydrogen bromide in glacial acetic acid (25 mL). The mixture was kept at room temperature for 24 h and then repeatedly concentrated with ethanol to give ethyl 3,4,6-tri-*O*-acetyl-2-deoxy-2-(3-phenylureido)-β-D-glucopyranoside (**7**; 0.45 g, 46%), m.p. 224–225° (from ethanol); $[\alpha]_{D}$ +10.5°, $[\alpha]_{578}$ +13°, $[\alpha]_{546}$ +15°, $[\alpha]_{436}$ +26°, $[\alpha]_{365}$ + 41.5° (*c* 1, chloroform); λ_{max} 270 and 239 nm (ε_{mM} 2.6 and 18.5); ν_{max} 3395–3230 (NH), 1735 (C=O ester), 1660 and 1635 (C=O urea), and 1560 cm^{-1} (NH). The ^{1}H- and ^{13}C-n.m.r. data are given in Tables I–III.

Anal. Calc. for $C_{21}H_{28}N_{2}O_{9}$: C, 55.75; H, 6.24; N, 6.19. Found: C, 56.14; H, 6.33; N, 6.16.

From the mother liquor of **7, 6** (0.2 g, 29%) was crystallised, m.p. 210–212° (from ethanol–water); $[\alpha]_{D}$ −27°, $[\alpha]_{578}$ −28°, $[\alpha]_{546}$ −34°, $[\alpha]_{436}$ −60°, $[\alpha]_{365}$ −98° (*c* 0.5, pyridine); λ_{max}^{EtOH} 269 and 239 nm (ε_{mM} 2.1 and 17.4); ν_{max} 3440–3100 (NH, OH), 1620 (C=O), and 1550 cm^{-1} (NH). ^{1}H-N.m.r. data [$(CD_3)_2SO$]: δ 8.41 (s, 1 H, N*H*Ph), 7.84–7.40 (m, 5 H, Ph), 6.06 (d, 1 H, $J_{2,NH}$ 6.7 Hz, N*H*CONHPh), 5.06

(m, 2 H, HO-3,4), 4.60 (t, 1 H, HO-6), 4.38 (d, 1 H, $J_{1,2}$ 7.8 Hz, H-1), 3.86–3.06 (m, 8 H, H-2,3,4,5,6,6′ and CH_2CH_3), and 1.09 (t, 3 H, CH_3). The ^{13}C-n.m.r. data are given in Table III.

Anal. Calc. for $C_{15}H_{22}N_2O_6$: C, 55.22; H, 6.80; N, 8.58. Found: C, 54.94; H, 6.90; N, 8.38.

(*b*) Conventional treatment of **6** (0.07 g, 0.2 mmol) with pyridine (0.3 mL) and acetic anhydride (0.45 mL) gave **7** (0.07 g, 74%).

A solution of **7** (0.4 g, 0.88 mmol) in hot ethanol (25 mL) was treated with ethanolic 0.1M sodium ethoxide (0.5 mL). On cooling, **6** (0.24 g, 83%) crystallized.

*Ethyl 2-deoxy-2-(3-methylureido)-β-*D*-glucopyranoside* (**8**). — Compound **8** (37%), prepared from 1,3,4,6-tetra-*O*-acetyl-2-deoxy-2-methylureido-α-D-glucopyranose[7] (**3**) as described for **1** and recrystallised from ethanol–water, had m.p. 211–213°; $[\alpha]_D$ −34°, $[\alpha]_{578}$ −35°, $[\alpha]_{546}$ −40°, $[\alpha]_{436}$ −67°, $[\alpha]_{365}$ −100° (*c* 0.5, water); λ_{max}^{EtOH} 269 nm (ε_{mM} 1.5); ν_{max} 3450–3240 (NH, OH), 1635 (C=O), and 1585 cm^{-1} (NH). ^{1}H-N.m.r. data [$(CD_3)_2SO$]: δ 5.30 (m, 2 H, N*H*CON*H*CH_3), 5.08 (d, 1 H, $J_{3,OH}$ 4.9 Hz, HO-3), 4.51 (t, 1 H, $J_{6,OH}$ 5.8 Hz, HO-6), 4.94 (d, 1 H, $J_{4,OH}$ 4.4 Hz, HO-4), 4.27 (d, 1 H, $J_{1,2}$ 8.0 Hz, H-1), 3.76–3.03 (m, 8 H, H-2,3,4,5,6,6′ and CH_2CH_3), 2.54 (d, 3 H, J_{NH,CH_3} 4.6 Hz, NHCH_3), and 1.09 (t, 3 H, $J_{CH_2CH_3}$ 7.4 Hz, CH_2CH_3). The ^{13}C-n.m.r. data are given in Table III.

Anal. Calc. for $C_{10}H_{20}N_2O_6$: C, 45.45; H, 7.63; N, 10.60. Found: C, 45.23; H, 7.58; N, 10.64.

*Ethyl 3,4,6-tri-*O*-acetyl-2-deoxy-2-methylureido-β-*D*-glucopyranoside* (**9**). — Conventional treatment of **8** (0.06 g, 0.22 mmol) with pyridine (0.3 mL) and acetic anhydride (0.45 mL) gave **9** (0.07 g, 80%), m.p. 220–221° (from ethanol); $[\alpha]_D$ +1°, $[\alpha]_{578}$ +3°, $[\alpha]_{546}$ +4°, $[\alpha]_{436}$ +8°, $[\alpha]_{365}$ +10.5° (*c* 0.6, chloroform); λ_{max}^{EtOH} 270 nm (ε_{mM} 1.7); ν_{max} 3400–3300 (NH) 1745 (C=O ester), 1645 (C=O urea), and 1590 cm^{-1} (NH). The ^{1}H- and ^{13}C-n.m.r. data are given in Tables I–III.

Anal. Calc. for $C_{16}H_{26}N_2O_9$: C, 49.23; H, 6.71; N, 7.17. Found: C, 49.51; H, 6.80; N, 7.11.

*2-Phenylamino-(3,4,6-tri-*O*-acetyl-1,2-dideoxy-α-*D*-glucofurano)[2,1-*d*]-2-oxazoline* (**11**). — (*a*) To a solution of **1** (4.0 g, 8.6 mmol) in dichloromethane (130 mL) was added anhydrous stannic chloride (4 mL). The mixture was kept for 48 h at room temperature and then buffered with saturated aq. sodium hydrogencarbonate. The organic layer was washed with water, dried ($MgSO_4$), and concentrated to dryness to give a homogeneous (t.l.c. and n.m.r.) amorphous solid (**11**; 2.9 g, 85%) that could not be recrystallised, and had m.p. 131–133°; $[\alpha]_D$ −6.5°, $[\alpha]_{578}$ −7°, $[\alpha]_{546}$ −9°, $[\alpha]_{436}$ −12°, $[\alpha]_{365}$ −17° (*c* 1, chloroform); λ_{max}^{EtOH} 275 and 247 nm (ε_{mM} 2.0 and 14.5); ν_{max} 3360 and 3200 (NH), 1760 (C=O), 1690 (C=N), 1595 (aromatic) and 1550 cm^{-1} (NH). The ^{1}H- and ^{13}C-n.m.r. data are given in Tables I–III.

Anal. Calc. for $C_{19}H_{22}N_2O_8$: C, 56.15; H, 5.46; N, 6.89. Found: C, 56.18; H, 5.45; N, 6.81.

(*b*) Compound **4**[11] was treated as in (*a*) to give **11** (82%).

*2-(4-Chlorophenylureido)-(3,4,6-tri-*O*-acetyl-1,2-dideoxy-α-*D*-glucopyrano)-[2,1-*d*]-2-oxazoline* (**12**). — Compound **12** (82%), obtained from 1,3,4,6-tetra-*O*-acetyl-2-[3-(4-chlorophenyl)ureido]-2-deoxy-α-D-glucopyranose[16] (**2**), as described above for **11**, had m.p. 98–99°; $[\alpha]_D$ +1°, $[\alpha]_{578}$ +1°, $[\alpha]_{546}$ +1°, $[\alpha]_{436}$ +0.5°, $[\alpha]_{365}$ +0.5° (*c* 0.9, chloroform); ν_{max} 3360, 3250, 3200, 3060 (NH), 1750 (C=O), 1670 (C=N), 1610 (aromatic), and 1560 cm^{-1} (NH). The ^1H- and ^{13}C-n.m.r. data are given in Tables I–III.

Anal. Calc. for $C_{19}H_{21}ClN_2O_8$: C, 51.77; H, 4.80; N, 6.35. Found: C, 51.72; H, 4.63; N, 6.57.

*2-Methylamino-(3,4,6-tri-*O*-acetyl-1,2-dideoxy-α-*D*-glucopyrano)[2,1-*d*]-2-oxazoline* (**13**). — Compound **13** (94%), obtained from **3**[7], as described above for **11**, had m.p. 123–124°; $[\alpha]_D$ +14°, $[\alpha]_{578}$ +15.5°, $[\alpha]_{546}$ +17°, $[\alpha]_{436}$ +27.5°, $[\alpha]_{365}$ +39.5° (*c* 0.9, chloroform); λ_{max}^{EtOH} 275 nm (ε_{mM} 0.5); ν_{max} 3500–3200 (NH), 1735 (C=O), and 1665 (C=N), and 1540 cm^{-1} (NH). The ^1H- and ^{13}C-n.m.r. data are given in Tables I–III.

Anal. Calc. for $C_{14}H_{20}N_2O_8$: C, 48.84; H, 5.86; N, 8.14. Found: C, 48.72; H, 5.93; N, 8.27.

Glycosylation of amino-oxazolines. — A solution of the 2-alkyl(aryl)amino-(3,4,6-tri-*O*-acetyl-1,2-dideoxy-α-D-glucopyrano)[2,1-*d*]-2-oxazoline in ethanol was heated under reflux for 7 h, then concentrated to dryness. The following compounds were prepared in this way.

Ethyl 3,4,6-tri-*O*-acetyl-2-deoxy-2-(3-phenylureido)-β-D-glucopyranoside (**7**, 67% from **11**), ethyl 3,4,6-tri-*O*-acetyl-2-deoxy-2-(3-methylureido)-β-D-glucopyranoside (**9**, 51% from **13**), and ethyl 3,4,6-tri-*O*-acetyl-2-[3-(4-chlorophenyl)ureido]-2-deoxy-β-D-glucopyranoside (**15**, 40% from **12**), m.p. 199–201° (from ethanol); $[\alpha]_D$ +8.5°, $[\alpha]_{578}$ +9°, $[\alpha]_{546}$ +9.5°, $[\alpha]_{436}$ +17.5°, $[\alpha]_{365}$ +31° (*c* 0.9, chloroform); ν_{max} 3400, 3370 (NH), 1740 (C=O ester), 1680, 1645 (C=O urea), 1595 (aromatic), and 1540 cm^{-1} (NH). The ^1H- and ^{13}C-n.m.r. data are given in Tables I–III.

Anal. Calc. for $C_{21}H_{27}ClN_2O_9$: C, 51.80; H, 5.59; N, 5.75. Found: C, 51.57; H, 5.64; N, 5.62.

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