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Note

Convenient syntheses of 2,3,5-tri-*O*-benzyl-arabino- and -ribofuranoses *via* their allyl glycosides

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2,3,5-Tri-*O*-benzyl-L-arabinofuranose[1] (**1**) and its D enantiomorph are available commercially and are useful intermediates in the synthesis of nucleoside antibiotics and various other derivatives of arabinofuranose[2-14]. Large amounts of **1** were required for work on the synthesis of β-L-arabinofuranosides[15]. A route to **1** *via* the allyl glycoside was found to be convenient and is now reported.

An improved version[16] of the original method[1] gave an overall yield of 48% of **1** in four steps from L-arabinose *via* methyl 2,3,5-tri-*O*-benzyl-L-arabinofuranoside. Difficulties encountered in the final, hydrolysis, step (~50% yield[16]) probably account for the loss of efficiency of the method[9,17]. It was suggested[1] that the degree of hydrolysis was thermodynamically limited despite the large excess of water. Our attempts to improve the yield of **1** by increasing the severity of the conditions of hydrolysis caused concomitant *O*-debenzylation.

Following the strategy of Gigg and Gigg[18], a route *via* the allyl glycoside **4** was investigated. Allyl ethers isomerise under basic conditions and the resulting prop-1-enyl ethers can be cleaved readily under mildly acidic[19] or other[20] conditions. We also envisaged that 2,3,4-tri-*O*-benzyl-L-arabinopyranose (**2**) and 2,3,5-tri-*O*-benzyl-D-ribofuranose (**3**) might be synthesised similarly, although in fact the hydrolysis of methyl 2,3,5-tri-*O*-benzyl-D-ribofuranoside is more efficient and an overall conversion of 52% from D-ribose was achieved[1].

An alternative approach *via* the benzyl tri-*O*-benzylglycosides yielded **1** (20%) and **2** (4%), but the overall conversion of D-ribose into **3** was negligible under the conditions used[21].

Fischer glycosidation of L-arabinose with allyl alcohol at 25° gave low yields of the allyl glycoside, probably because of the low solubility of the sugar. However, reaction at 40° in the presence of sulphuric acid gave ~85% of **4** (α,β-ratio 2.2:1); the use of an acid resin catalyst was less successful. Benzylation[22] of **4**, using benzyl chloride and

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sodium hydride in methyl sulphoxide, proceeded without allyl→prop-1-enyl isomerisation. Other benzylation conditions (*e.g.*, the use of such bases as methanesulphinylmethanide derived from potassium *tert*-butoxide[23] or sodium hydride[24]) also failed to cause such isomerisation[19]. The allyl tri-*O*-benzylarabinosides **5** (isolated in ~90% yield) were isomerised[19] to **6** (~95%), using potassium *tert*-butoxide in methyl sulfoxide. Mild acid hydrolysis[19] of the prop-1-enyl glycosides **6** gave known[1,25] 2,3,5-tri-*O*-benzyl-β-L-arabinofuranose (**1**, 95%). The overall yield of **1** from L-arabinose was ~70%. The synthesis could be carried out on a 100-g scale without the need to isolate any of the intermediates.

As expected, more vigorous or prolonged reaction in the Fischer glycosidation step gave mainly the allyl α,β-pyranosides **7** (~90%). The β anomer (**7**β) was obtained crystalline and further characterised as the crystalline 3,4-*O*-isopropylidene derivative **8**, prepared using acetone and sulphuric acid[25]. Compound **7**β was converted by the standard methods[19,20] used above for **1**, *via* **9** and **10**, into the known[16] 2,3,4-tri-*O*-benzyl-β-L-arabinopyranose (**2**, 77% from L-arabinose).

Fischer glycosidation conditions were also established for the conversion of D-ribose into a mixture that contained mainly the allyl α,β-D-ribofuranosides (**11**, ~95%; α,β-ratio ~1:3.4). Crude **11** was converted[19,20] into 2,3,5-tri-*O*-benzyl-α,β-D-ribofuranose (**3**, 76% from D-ribose), *via* **12** and **13**.

^{13}C-N.m.r. spectroscopy is of particular importance for the establishment of the ring size[26–31] of sugar derivatives. The characteristic resonance in glycofuranosides due to C-4 appears at δ ~85. The C-2/5 resonances for pyranosides are confined to the region below δ ~75, whereas those for furanosides lie above this value, and these differences were of particular value in ascertaining the purity of the mixtures obtained in the Fischer glycosidation step. The assignments of ^{13}C resonances to individual anomers were made on the basis[30,32] of the chemical shifts of the C-1 resonances. The allyl glycosides **4**, **5**, **7**, **9**, **11**, and **12** had resonances at δ ~69, ~120, and ~135, which are characteristic of the allyl group, and the prop-1-enyl glycosides **6**, **10**, and **13** were identified by the resonances at δ ~60, ~140, and ~141. For the 3,4-*O*-isopropylidene derivative **8**, the chemical shift (109.21 p.p.m.) of the acetal carbon resonance and the $\Delta\delta$ value (1.98 p.p.m.) of the resonances of the acetal methyl carbons were in the range expected[33–35] for a 1,3-dioxolane ring *cis*-fused to a pyranoid ring.

EXPERIMENTAL

M.p.s. were determined in capillary tubes with a Thomas–Hoover apparatus and are uncorrected. Optical rotations were measured using a Perkin–Elmer 241 polarimeter. N.m.r. spectra were obtained with Jeol FX90Q (^{1}H, 89.6 MHz; ^{13}C, 22.5 MHz) or Bruker WH-400 spectrometers (^{1}H, 400.13 MHz; ^{13}C, 100.0 MHz). F.a.b.-mass spectra were recorded with a VG ZAB-1F spectrometer, using a thioglycerol–oxalic acid matrix. Microanalyses were performed by the microanalytical services of the Bourne Laboratory. T.l.c. was performed on Polygram Sil G (Machery–Nagel) with detection by charring with sulphuric acid. Flash chromatography was conducted as described[36]. Evaporations *in vacuo* were conducted at $<35°$ (bath). Solids and syrups were dried at 0.4 mmHg and 25° for 6–24 h. All other dry reagents were prepared by standard methods[37]. Sodium hydride, supplied as a 50% dispersion in mineral oil, was freed from the oil by repeated washing with dry ether under N_2.

*Allyl α,β-*L*-arabinofuranoside* (**4**). — Conc. sulphuric acid (0.52 mL) was added dropwise to a vigorously stirred, ice-cooled, dry suspension of finely powdered L-arabinose (5 g, 33 mmol) and granulated calcium sulphate (2.5 g) in allyl alcohol (70 mL, 1 mol). The mixture was stirred vigorously at 40° with the exclusion of moisture, and the reaction was monitored by t.l.c. (ethyl acetate–1-propanol–water, 5:3:1). Within ~6 h, the arabinose (R_F 0.57) was converted into products with R_F 0.81 and 0.86 which, on further reaction, were converted into products with R_F 0.71 and 0.75. The mixture was filtered, the insoluble material was washed with fresh, dry allyl alcohol (50 mL), and the filtrate and washings were combined and passed through a column of Amberlite IR-45 (HO^-) resin packed in fresh, dry allyl alcohol (50 mL). The resin was washed with fresh allyl alcohol (50 mL), and the combined eluate and washings were concentrated *in vacuo*. The residue was dried (10 h) to give **4** as a pale-yellow syrup (5.55 g, 88%) which was used directly in the next step. ^{13}C-N.m.r. data (D_2O, internal 1,4-dioxane, 67.80 p.p.m.): α anomer, δ 109.11 (C-1), 83.56 and 78.95 (C-2,3), 86.17 (C-4), 63.73 (C-5), 71.21 (OCH_2), 136.24 ($-CH=$), and 120.70 ($=CH_2$); β anomer, 102.88 (C-1), 77.39 and 78.95 (C-2,3), 84.54 (C-4), 65.68 (C-5), other resonances as for the α anomer.

*Allyl 2,3,5-tri-*O*-benzyl-α,β-*L*-arabinofuranoside* (**5**). — A solution of dry **4** (3 g, 15.8 mmol) in dry methyl sulfoxide (9 mL) was added dropwise to a vigorously stirred, ice-cooled suspension of sodium hydride (1.7 g, 70 mmol) in methyl sulfoxide (12 mL) under nitrogen. The mixture was stirred for 1 h at 25°, dry benzyl chloride (8.0 mL, 70 mmol) was then added dropwise, and stirring was continued overnight at 25° (in larger scale reactions, very slow addition of benzyl chloride was required to prevent charring). Methanol (3 mL) was added to destroy the excess of sodium hydride, the mixture was diluted with water and extracted with ether (75 mL total), and the ether extracts were combined, dried ($MgSO_4$), and concentrated *in vacuo* to give crude **5** as a pale-yellow syrup (6.7 g, 93%) which was used directly in the next step.

Flash chromatography (light petroleum–ethyl acetate, 3:1) of a portion of the product gave **5β** as a syrup, R_F 0.6, $[\alpha]_D^{25}$ +50° (*c* 1, dichloromethane). ^{13}C-N.m.r. ($CDCl_3$): δ 100.02 (C-1), 84.48 and 81.49 (C-2,3), 89.16 (C-4), 70.63 and 68.73 (C-5 and

$OCH_2CH=CH_2$), 72.71, 72.84, and 73.43 (benzyl CH_2), 128.4–130.4 (phenyl CH), 138.4–138.9 (phenyl C), 120.70 ($=CH_2$), and 136.24 ($=CH$). F.a.b.-mass spectrum: m/z 483 ($M + Na^+$).

Anal. Calc. for $C_{29}H_{32}O_5$: C, 75.6; H, 7.5. Found: C, 75.4; H, 7.5.

Prop-1-enyl 2,3,5-tri-O-benzyl-α,β-L-arabinofuranoside (**6**). — A solution of dry **5** (1.0 g, 2.2 mmol) in dry methyl sulfoxide (10 mL) was heated with potassium *tert*-butoxide (1.26 g) for 1 h at 95–100° (bath), then cooled, diluted with water, and extracted with ether (100 mL total). The combined extracts were washed with saturated aqueous NaCl, dried ($MgSO_4$), and concentrated *in vacuo* to give crude **6** as a pale-brown syrup (0.95 g, 93%) which was used directly in the next step.

Flash chromatography (light petroleum–ethyl acetate, 3:1) of a portion of the syrup gave an analytical sample of **5**α,β, R_f 0.77. ^{13}C-N.m.r. data ($CDCl_3$): major anomer: δ 106.07 (C-1), 83.50 and 81.36 (C-2,3), 87.86 (C-4), 64.42 (C-5), 72.09, 72.13, and 73.43 (benzyl CH_2), 126.0–130.3 (phenyl CH), 137.6–138.5 (phenyl C), 60.36 (CH_3), 104.27 ($=CH$), 141.84 ($OCH=$). F.a.b.-mass spectrum: m/z 483 ($M + Na^+$).

Anal. Calc. for $C_{29}H_{32}O_5$: C, 75.6; H, 7.5. Found: C, 75.3; H, 7.4.

2,3,5-Tri-O-benzyl-β-L-arabinofuranose (**1**). — A solution of **6** (0.5 g, 1.1 mmol) in acetone was boiled under reflux with 0.5M sulphuric acid (0.6 mL), then cooled, diluted with aqueous Na_2CO_3, and extracted with dichloromethane (30 mL total). The combined extracts were washed with saturated aqueous NaCl, dried (Na_2SO_4), and concentrated *in vacuo* to give **1**, which crystallised on storage (0.42 g, 95%). A solution of the remaining syrup in ethyl acetate was treated with activated carbon, filtered through Celite, and diluted with pentane. The product was recrystallised from ethyl acetate–light petroleum to give **1** as colourless needles, m.p. 84–85°, $[\alpha]_D^{20}$ +4.4° (*c* 1.3, dichloromethane); lit.[16] for the β anomer, m.p. 88–89°, $[\alpha]_D^{20}$ +6.5° (dichloromethane). ^{13}C-N.m.r. data ($CDCl_3$): α anomer, 101.11 (C-1), 82.82 and 81.78 (C-2,3), 86.83 (C-4), 70.81 (C-5), 71.76–73.52 (benzyl CH_2), 127.5–129.8 (phenyl CH), 137.3–138.0 (phenyl C).

Allyl β-L-arabinopyranoside (**7**). — Conc. sulphuric acid (0.5 mL) was added dropwise to a vigorously stirred, ice-cooled, dry suspension of finely powdered L-arabinose (5 g, 33 mmol) and granulated calcium sulphate (2.5 g) in allyl alcohol (70 mL, 1 mol). The mixture was stirred vigorously for 24 h at 80° with the exclusion of moisture. T.l.c. (5:3:1 ethyl acetate–1-propanol–water) of the mixture then showed that the arabinose (R_f 0.57) had been converted into products with R_f 0.71 and 0.75 contaminated by a small amount of the furanosides (R_f 0.81 and 0.86). The reaction was stopped and the products were isolated as described for **4**. A solution of the resulting pale-brown syrup (5.8 g, 92%) in ethanol was treated with activated carbon, filtered through Celite, and ice-cooled to give **7**β as needles, m.p. 118–120°, $[\alpha]_D^{20}$ +179° (*c* 1, water). ^{13}C-N.m.r. data (D_2O, internal 1,4-dioxane): δ 100.22 (C-1), 70.45, 71.83, and 72.83 (C-2,3,4), 65.03 (C-5), 70.43 (OCH_2), 135.91 ($=CH$), 120.44 ($=CH_2$). F.a.b.-mass spectrum: m/z 213 ($M + Na^+$).

Anal. Calc. for $C_8H_{14}O_5$: C, 50.5; H, 7.4. Found: C, 50.3; H, 7.5.

Allyl 3,4-O-isopropylidene-β-L-arabinopyranoside (**8**). — Crystalline **7** was treated with dry acetone and sulphuric acid, as described[25] for the synthesis of methyl 3,4-*O*-iso-

propylidene-β-D-arabinopyranoside, to give crude **8** as a syrup which crystallised on storage. Flash chromatography (1:1 light petroleum–ethyl acetate) gave **8**, R_F 0.55, m.p. 71°, $[\alpha]_D^{20}$ +209° (*c* 1.1, dichloromethane). ^{13}C-N.m.r. data ($CDCl_3$): δ 133.64 (CH=), 117.82 (CH_2=), 109.21 (CMe_2), 96.88 (C-1), 76.05, 72.99, and 70.07 (C-2,3,4), 68.66 (C-5), 59.76 (OCH_2), 27.95, 25.97 (acetal CH_3). F.a.b.-mass spectrum: *m/z* 253 ($M + Na^+$).

Anal. Calc. for $C_{11}H_{18}O_5$: C, 57.4; H, 7.9. Found: C, 57.3; H, 7.6.

Flash chromatography (light petroleum–ethyl acetate, 3:1) gave, in addition to **8**, 1,2:3,4-di-*O*-isopropylidene-β-L-arabinopyranose, m.p. 41–42°, $[\alpha]_D^{20}$ +4.2° (*c* 0.95, water); lit.[38] m.p. 41.5–43°, $[\alpha]_D^{20}$ +5.4° (water).

Allyl 2,3,4-tri-O-benzyl-β-L-arabinopyranoside (**9**). — Crystalline **7** (3 g, 15.8 mmol) was benzylated using sodium hydride, methyl sulfoxide, and benzyl chloride, as described to obtain **5**, to give crude **9** as a pale-yellow syrup (6.9 g, 95%). Flash chromatography (2:1 light petroleum–ethyl acetate) of a portion gave syrupy **9**, $[\alpha]_D^{20}$ +47° (*c* 1.1, dichloromethane). ^{13}C-N.m.r. data ($CDCl_3$): δ 96.83 (C-1), 77.24, 76.40, and 74.00 (C-2,3,4), 60.44 (C-5), 71.7–73.5 (benzyl CH_2), 126.8–128.4 (phenyl CH), 138.2–138.8 (phenyl C), 68.32 ($OCH_2CH=CH_2$), 117.70 ($=CH_2$), 134.00 (=CH). F.a.b.-mass spectrum: *m/z* 483 ($M + Na^+$).

Anal. Calc. for $C_{29}H_{32}O_5$: C, 75.6; H, 7.5. Found: C, 75.8; H, 7.6.

2,3,4-Tri-O-benzyl-L-arabinopyranose (**2**). — Crude **9** (1 g, 2.2 mmol) was isomerised using potassium *tert*-butoxide in methyl sulfoxide, as described to obtain **6**, to give crude syrupy **10** (0.94 g, 95%). Crude **10** (0.5 g, 1.1 mmol) was hydrolysed using aqueous sulphuric acid in acetone, as described for the preparation of **1**, to give crude **2** as a pale-brown syrup (0.41 g, 93%), a solution of which in di-isopropyl ether was treated with activated carbon and then concentrated to give an α,β-mixture. On storage, **2**β crystallised with m.p. 76–78° (from di-isopropyl ether), $[\alpha]_D^{20}$ +56° (*c* 1.2, dichloromethane); lit.[16] m.p. 71–79°, $[\alpha]_D^{20}$ +93.2° (9:1 1,4-dioxane–water, 3 min); lit.[16] m.p. 83–86° (from cyclohexane), $[\alpha]_D^{20}$ +66.5→+51.1° (dichloromethane). ^{13}C-N.m.r. data ($CDCl_3$): α anomer, δ 92.04 (C-1), 76.65 and 76.49 (C-2,3), 72.90 (C-4), 60.84 (C-5), 73.6–71.5 (benzyl CH_2), 126.9–129.7 (phenyl CH), 137.5–138.4 (phenyl C); β anomer, 93.97 (C-1), 76.36 and 75.48 (C-2,3), 72.5 (C-4), 58.60 (C-5), other resonances as for α anomer.

Allyl α,β-D-ribofuranoside (**11**). — D-Ribose (5 g, 33 mmol) and allyl alcohol were reacted together, as described for the preparation of **2**, but for 6 h at 25°. Crude **11** was obtained as a syrup (6.0 g, 96%) which was used directly in the next step. T.l.c. (ethyl acetate–1-propanol–water, 5:3:1) revealed this mixture to contain mainly **11** (R_F 0.81) together with ~10% of a component with R_F 0.77, but no D-ribose (R_F 0.44). ^{13}C-N.m.r. data [$(CD_3)_2SO$, external Me_4Si]: α anomer, δ 103.79 (C-1), 72.18 and 71.73 (C-2,3 and allyl OCH_2), 86.88 (C-4), 63.99 (C-5), 136.5 (=CH), 120.50 ($=CH_2$); β anomer, 108.60 (C-1), 76.87 (C-2), 73.42 and 71.21 (C-3 and allyl OCH_2), 85.32 (C-4), 65.42 (C-5), 135.9 (=CH), 120.96 ($=CH_2$).

Longer reaction times and higher temperatures led to increased proportions of the minor components which spectral data indicated to be the anomeric D-ribopyranosides. ^{13}C-N.m.r. data (D_2O; internal 1,4-dioxane, at 67.80 p.p.m.): α anomer, δ 100.28

(C-1), 70.0 (C-2,3 and allyl OCH_2), 67.37 (C-4), 63.86 (C-5), 135.85 (=CH), 120.70 ($=CH_2$); β anomer, δ 101.84 (C-1), 69.26 (C-4), 65.29 (C-5), 136.37 (=CH), 120.31 ($=CH_2$).

*Allyl 2,3,5-tri-*O*-benzyl-α,β-*D*-ribofuranoside* (**12**). — Crude **11** (3.0 g, 15.8 mmol) was benzylated using sodium hydride, methyl sulfoxide, and benzyl chloride, as described to obtain **5**, to give crude **12** as a yellow syrup (6.4 g, 89%), a portion of which was purified by flash chromatography (1:1 light petroleum–ethyl acetate). ^{13}C-N.m.r. data ($CDCl_3$): δ 134.13 (CH=), 127.5–128.67 (phenyl C), 117.22 (CH_2=), 104.46 (C-1), 80.52, 79.81, 78.57 (C-2,3,4), 73.14, 72.43, 72.33, 71.37 (C-5 and benzyl CH_2), 68.25 (OCH_2). F.a.b.-mass spectrum: *m/z* 483 ($M + Na^+$).

Anal. Calc. for $C_{29}H_{32}O_5$: C, 75.6; H, 7.5. Found: C, 75.4; H, 7.8.

*Tri-*O*-benzyl-α,β-*D*-ribofuranose* (**3**). — Compound **12** (1.0 g, 2.2 mmol) was isomerised using potassium *tert*-butoxide in methyl sulfoxide, as described to obtain **5**, to give crude syrupy **13** (0.94 g, 95%).

Crude **13** (0.5 g, 1.1 mmol) was hydrolysed using aqueous sulphuric acid in acetone, as described for the preparation of **1**, to give crude **3** as a pale red-brown syrup (0.42 g, 94%). Flash chromatography, as described above, gave **3**α,β with $[\alpha]_D^{20}$ +26° (*c* 1.2, dichloromethane); lit.[1] $[\alpha]_D^{20}$ +37.0° (1,4-dioxane).

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