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2-Deoxy-L-ribose from an L-*arabinono*-1,5-lactone

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Abstract—A practical synthesis of 2-deoxy-L-ribose from L-arabinose depends on the efficient reduction by iodide of a triflate α to a lactone. The X-ray crystal structure of 3,4-*O*-isopropylidene-L-*arabinono*-1,5-lactone is reported. © 2002 Published by Elsevier Science Ltd.

1. Introduction

The majority of antiviral drugs are nucleoside analogues. L-Nucleosides are recognised by virus-encoded or bacterial enzymes–but not by host enzymes–and have a better profile of good antiviral activity combined with minimal host toxicity.[1] 3TC (Lamivudine) was the first L-nucleoside approved for use in combination therapy against HIV and HBV.[2] Further examples of L-nucleosides, such as L-thymidine, possessing potent antiviral activity have identified a need for an efficient synthesis of homochiral 2-deoxy-L-ribose **2** from cheap starting materials.

Although L-ascorbic acid **3** has been used as a starting material,[3,4] the majority of syntheses of **2** start from L-arabinose **1** in which all that is needed is the removal of the OH function at C-2 (Scheme 1). The classic procedure via the formation of L-arabinal[5] is experimentally challenging on a large scale. In all the other syntheses, it is necessary to form either a protected pyranose **1p** or furanose **1f** of arabinose in which only C-2 OH is free. Access to suitably protected derivatives of **1f** usually requires a number of manipulations,[6] and almost all the procedures utilise radical deoxygenations in the reductive step.[7,8] In contrast arabinopyranose derivatives **1p** with C-2 OH free are relatively easily formed. However, nucleophilic substitution of leaving groups at C-2 of arabinose is fraught with problems[9] and again it is necessary to use Barton radical deoxygenation procedures to produce protected pyranose derivatives of deoxyribose **2p**.[10] A practical procedure for the synthesis of 2-deoxy-L-ribose **2** from a readily accessible arabinopyranose derivative that avoids radical deoxygenation is needed.

2. Results and discussion

This paper reports a procedure in which the C-2 OH group of an *arabinono*-lactone is converted to a triflate and subsequently removed by reduction by iodide.

The kinetic monoacetonide of L-arabinose **4** is readily available in high yield by treatment of arabinose with dimethoxypropane in DMF in the presence of a catalyst of *p*-toluenesulfonic acid.[11] Treatment of the lactol **4** with bromine in dichloromethane in the presence of sodium carbonate formed the crystalline lactone **5** in 75% yield (Scheme 2), $[\alpha]_D^{21}$ −29.2 (*c* 1.01, $CHCl_3$), $[\alpha]_D^{21}$ −91.5 (*c* 1.93, MeOH); the oxidation previously reported by silver carbonate on Celite proceeded in a much lower yield and with a very different specific rotation $[\alpha]_D$ −1 (*c* 3.0, $CHCl_3$).[12] The 1,5-lactone **5** has also been formed from treatment of L-arabinono-1,4-lactone in 16% yield as a foam;[13] although the NMR data for **5** closely matches the literature data, again the specific rotation of the sample was very different from that previously reported, $[\alpha]_D^{21}$ −9.8 (*c* 1.8, MeOH).[14] Accordingly the structure of the hydroxylactone **5** was firmly established by X-ray crystallographic analysis (Fig. 1). The lactone ring in **5** adopts a 'boat' conformation with C-2 and C-5 occupying the 'bow' positions and the C-2 OH. The hydroxyl group occupies an axial

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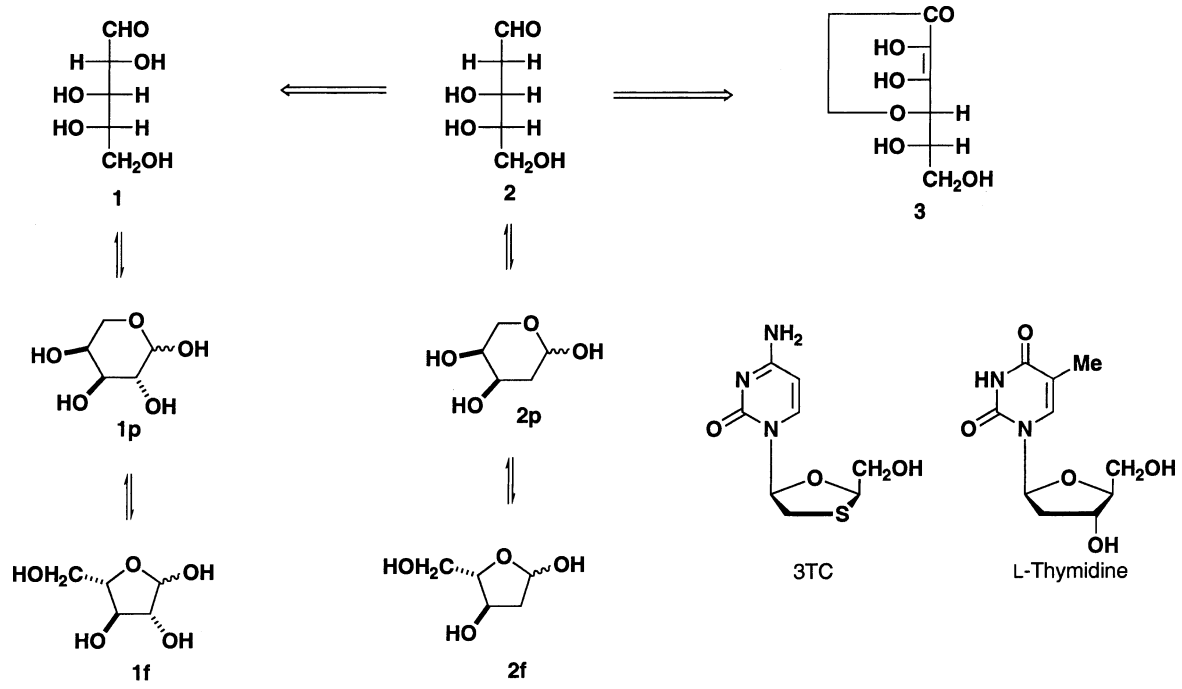
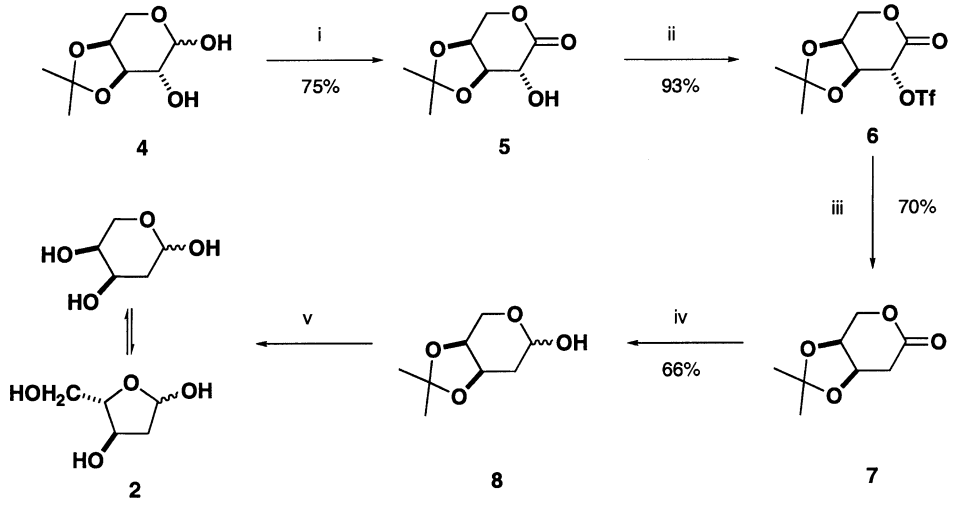


Scheme 1.



Scheme 2. *Reagents and conditions*: (i) Br_2, Na_2CO_3, CH_2Cl_2; (ii) Tf_2O, pyridine, CH_2Cl_2; (iii) $LiI{\cdot}3H_2O$, MeCOEt, 80°C; (iv) DIBALH, CH_2Cl_2, −50°C; (v) $CF_3COOH{:}H_2O$.

position. The molecules are linked by hydrogen bonds formed between the hydroxyl group and the carbonyl O of a neighbouring molecule (O(2)···O(1)′ 2.722(2) Å) to form infinite chains running parallel to the crystallographic *b* axis (Fig. 2).

Esterification of free hydroxyl group in **5** by treatment with trifluoromethanesulfonic (triflic) anhydride in dichloromethane in the presence of pyridine gave the corresponding triflate in 93% yield. Reaction of the triflate **6** with lithium iodide hydrate in butanone gave the deoxygenated lactone **7** in 70% yield. The reduction[15] may proceed by S_N2 displacement of the triflate group in **6** by iodide, followed by further nucleophilic attack by iodide on the *ribo*-iodo function to produce iodine and an enolate which on protonation gives the deoxylactone **7** (Scheme 3). The enantiomer of **7** has been previously prepared by the oxidation[16] of the enantiomer of the lactols **8**.[17]

Reduction of the deoxylactone **7** with diisobutylaluminum hydride (DIBALH) gave the lactols **8** in 66% isolated yield, with no over reduction. Subsequent treatment of **8** with 0.4% trifluoroacetic acid in water gave efficient deprotection to the target 2-deoxy-L-ribose **2**. It is likely that on a larger scale, the DIBALH-hydrolysis sequence will proceed in an overall excellent yield.

3. Conclusion

2-Deoxy-3,5-di-*O*-toluoyl-α-D-*erythro*-pentofuranosyl chloride **10** is one of the most commonly used precur-

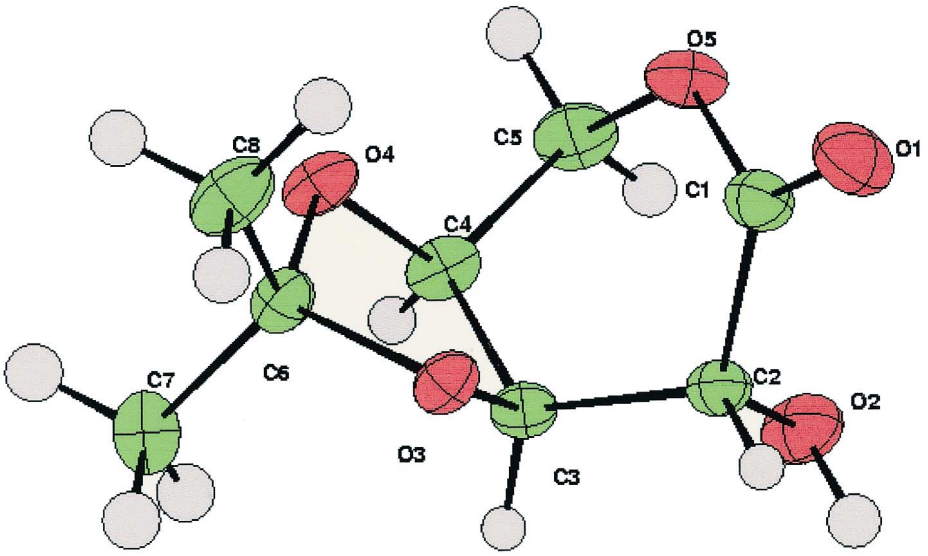


Figure 1. Thermal ellipsoid plot (ORTEP-3[23]) at 40% probability of 3,4-*O*-isopropylidene-L-*arabinono*-1,5-lactone **5**.

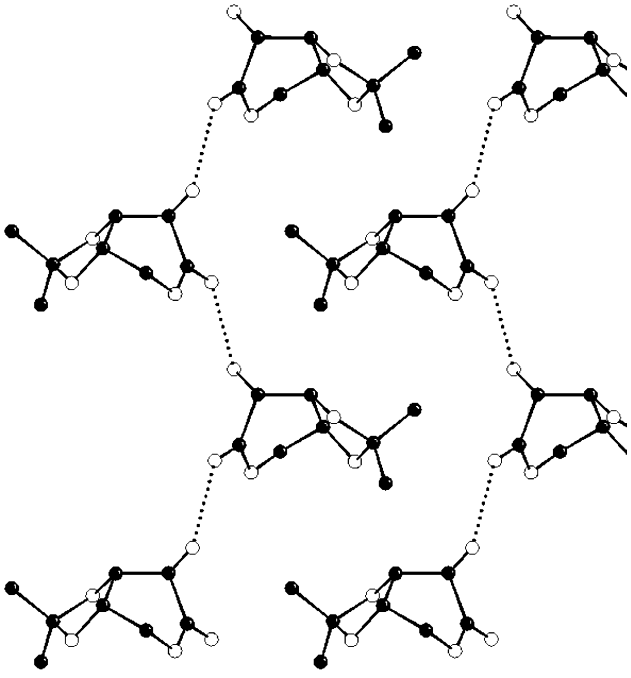


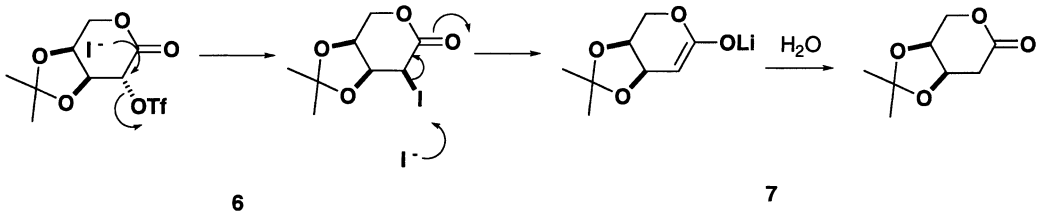
Figure 2. Crystal stacking of 3,4-*O*-isopropylidene-L-*arabinono*-1,5-lactone **5**.

sors for the synthesis of a wide variety of deoxyribonucleosides usually with an excellent selectivity for the formation of the required β-anomers **11** (Scheme 4) by Vorbrüggen and other coupling procedures. The α-chlorofuranose form **10** is efficiently established from the unprotected 2-deoxy-D-ribose **9**.[18]

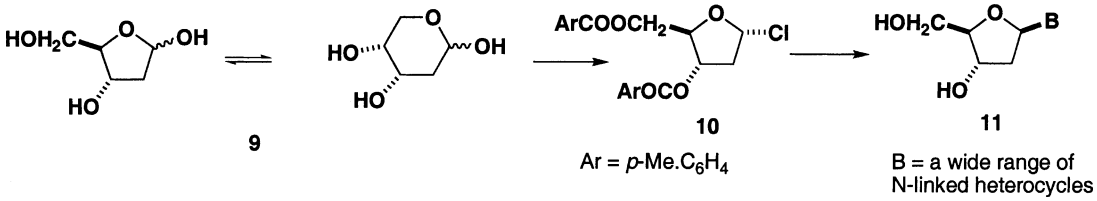
This paper reports an efficient but non-optimised procedure for the synthesis of 2-deoxy-L-ribose **2** which is an intermediate likely to be of value in the generation of new L-nucleosides by analogous procedures to those indicated for the D-series in Scheme 4.

4. Experimental

Proton nuclear magnetic resonance spectra (δ_H) were recorded on a Bruker DPX 400 (400 MHz) or Bruker DPX 200 (200 MHz) spectrometer, and were calibrated according to the chemical shift of residual protons in the deuterated solvent. ^{13}C NMR spectra (δ_C) were recorded on a Bruker DPX 400 (100.6 MHz) or Bruker DPX 200 (50.3 MHz) spectrometer and were calibrated using chemical shift of the deuterated solvent. Chemical shifts (δ) are quoted in ppm and coupling constants (J) in Hz. The following abbreviations are used to denote multiplicities: s, singlet; d, doublet; dd, double-doublet; ddd, double-double-doublet; t, triplet; q, quartet; dt, double-triplet; quint, quintuplet; m, multiplet; br, broad; a, apparent. Infrared spectra were recorded on a Perkin–Elmer 1750 FT IR spectrophotometer using thin films on NaCl plates and peaks are given in cm^{-1}. Low-resolution mass spectra were recorded either on a VG platform atmospheric pressure chemical ionisation (APCI), Micromass GCT spectrometer (GC–MS) or a Micromass LCT spectrometer (ESI). High-resolution mass spectra (HRMS) were recorded on a Micromass VG Autospec 500 OAT (CI, NH_3) spectrometer. Optical rotations were measured on a Perkin–Elmer 241 polarimeter with a path length of 1 dm, concentrations (c) are quoted in g/100 ml. Elemental analyses were performed by the microanalysis service of the Inorganic Chemistry Laboratory (Oxford). Melting points (mp) were measured on a Kofler hot-block apparatus and are uncorrected. Thin-layer chromatography (TLC) was carried out on aluminium sheets coated with $60F_{254}$ silica from Merck, and plates were developed using either a spray of vanillin (8 g/l) in 95% ethanol containing 1% (v/v) sulfuric acid or 0.2% w/v cerium(IV)sulfate and 5% ammonium molybdate in 2 M sulfuric acid and subsequent heating. Flash chromatography was carried out using Sorbsil C60 40/60 silica. Solvents were used as supplied (HPLC grade) and commercially available reagents were used as supplied. Hexane refers to petroleum ether boiling in the range 60–80°C. 3,4-*O*-



Scheme 3.



Scheme 4.

Isopropylidene-L-arabinopyranose **4** was prepared by the kinetic acetonation of L-arabinose as previously described.[12]

4.1. 3,4-*O*-Isopropylidene-L-*arabinono*-1,5-lactone 5

Bromine (10.3 mL, 0.2 mol) was added dropwise over 30 min to a stirred suspension of sodium carbonate (21.2 g, 0.2 mol) in a solution of 3,4-*O*-isopropylidene-L-arabinopyranose **4** (25 g, 0.13 mol) in dichloromethane (160 mL) at room temperature. The reaction mixture was stirred for a further 5 h when TLC analysis (ethyl acetate:hexane, 9:1) indicated a major product (R_f 0.46). Nitrogen was bubbled through the solution for 30 min to remove excess unreacted bromine; the suspension filtered through celite (dichloromethane as eluant). The filtrate was washed with sodium thiosulfate solution (0.5 M $Na_2S_2O_3$, 200 mL); the aqueous extract was re-extracted with dichloromethane (3×300 mL). The combined organic extracts were dried with magnesium sulfate, filtered and concentrated in vacuo to yield a yellow oil. This oil was purified using column chromatography (ethyl acetate:hexane, 2:3, preabsorbed from ethyl acetate) to yield 3,4-*O*-isopropylidene-L-*arabinono*-1,5-lactone **5** (18.3 g, 75%) as a white crystalline solid; mp 94–95°C {Lit.[12] 95–97°C}; $[\alpha]_D^{21}$ −29.2 (*c* 1.01, $CHCl_3$) {Lit.[12] $[\alpha]_D$ −1 (*c* 3.0, $CHCl_3$)}; ν_{max} (film): 3429 (OH), 1754 (C=O) cm^{-1}; δ_H ($CDCl_3$, 400 MHz): 1.39, 1.53 (2×3H, 2×s, $C(CH_3)_2$), 3.38 (1H, d, $J_{OH,2}$ 3.46 Hz, 2-OH), 4.13 (1H, dd, $J_{5,5'}$ 11.38 Hz, $J_{4,5'}$ 8.24 Hz, H-5′), 4.36 (1H, dd, $J_{2,3}$ 6.19 Hz, $J_{3,4}$ 7.23 Hz, H-3), 4.44 (1H, dd, $J_{2,OH}$ 3.36 Hz, $J_{2,3}$ 5.95 Hz, H-2), 4.51 (1H, ddd, $J_{4,5}$ 5.24 Hz, $J_{4,5'}$ 7.8 Hz, $J_{3,4}$ 7.23 Hz, H-4), 4.59 (1H, dd, $J_{5,5'}$ 11.06 Hz, $J_{4,5}$ 5.11 Hz, H-5); δ_C ($CDCl_3$, 100 MHz): 24.8, 27.1 ($C(\underline{C}H_3)_2$), 67.3 (C-5), 70.5 (C-4), 70.7 (C-2), 77.7 (C-3), 112.2 ($\underline{C}(CH_3)_2$), 172.1 (C-1); *m*/*z* (APCI +ve): 189 ($M+H^+$, 100%). Found: C, 51.06; H, 6.42; $C_8H_{12}O_5$ requires C, 51.06; H, 6.43.

4.2. 3,4-*O*-Isopropylidene-2-*O*-trifluoromethanesulfonyl-L-*arabinono*-1,5-lactone 6

Triflic anhydride (14.1 mL, 0.08 mol) was added to a stirred solution of the hydroxylactone **5** (12.98 g, 0.07 mol) in dichloromethane (200 mL) and pyridine (6.79 mL, 0.08 mol) maintaining the temperature between −10 and −20°C over 30 min. After 2.5 h, TLC analysis (ethyl acetate:hexane, 2:3) indicated a new product (R_f 0.43). The reaction mixture was diluted with dichloromethane (400 mL), washed with water (350 mL) and the aqueous layer further extracted with dichloromethane (3×150 mL). The combined organic extracts were dried (magnesium sulfate), filtered and concentrated in vacuo (co-evaporated with toluene); the residue was purified using flash chromatography (ethyl acetate:hexane, 2:3) to afford the triflate **6** (21.0 g, 93%) as a white solid, mp 88–89°C; $[\alpha]_D^{20}$ +24.5 (*c* 1.04, $CHCl_3$); ν_{max} (film): 1773 (C=O) cm^{-1}; δ_H ($CDCl_3$, 400 MHz): 1.41, 1.53 (2×3H, 2×s, $C(CH_3)_2$), 4.27 (1H, ddd, H-5′), 4.65 (1H, m, H-3, H-4, H-5), 5.23 (1H, d, $J_{2,3}$ 5.77 Hz, H-2); δ_C ($CDCl_3$, 100 MHz): 24.4, 26.4 ($C(\underline{C}H_3)_2$), 67.0 (C-5), 70.1 (C-4), 74.4 (C-3), 80.0 (C-2), 113.1 ($\underline{C}(CH_3)_2$), 163.1 (C-1); *m*/*z* (APCI +ve): 321 ($M+H^+$, 75%). Found: C, 33.64; H, 3.42; $C_9H_{11}F_3O_7S$ requires C, 33.75; H, 3.46.

4.3. 2-Deoxy-3,4-*O*-isopropylidene-L-*ribono*-1,5-lactone 7

The triflate **6** (5.19 g, 0.016 mol) and lithium iodide trihydrate (19 g, 0.10 mol) were dissolved in butanone (140 mL) and the reaction mixture heated at 80°C. After 24 h, TLC analysis (ethyl acetate:hexane, 2:1) indicated no remaining starting material (R_f 0.53) and a single new product (R_f 0.24). The reaction mixture was concentrated in vacuo, diluted with ethyl acetate (200 mL) and washed with 0.5 M sodium thiosulfate solution (150 mL) to remove the iodine. The aqueous extracts were further extracted with ethyl acetate (2×80 mL); the combined organic extracts were dried (magne-

sium sulfate), filtered and concentrated in vacuo to yield a light orange solid. This residue was purified using column chromatography (ethyl acetate:hexane, 2:1) to yield 2-deoxy-3,4-*O*-isopropylidene-L-*ribono*-1,5-lactone **7** (1.93 g, 70%) as a white solid; mp 118–120°C [Lit.[19] for enantiomer 118–119°C]; $[\alpha]_D^{23}$ +148.1 (*c* 1.03, $CHCl_3$) [Lit.[19] for enantiomer $[\alpha]_D^{22}$ −146.8 (*c* 0.62, $CHCl_3$)]; ν_{max} (film): 1742 (C=O) cm^{-1}; δ_H ($CDCl_3$, 400 MHz): 1.34, 1.45 (2×3H, 2×s, $C(CH_3)_2$), 2.53 (1H, dd, $J_{2,2'}$ 15.96 Hz, $J_{2',3}$ 3.76 Hz, H-2′), 2.53 (1H, dd, $J_{2,2'}$ 15.95 Hz, $J_{2,3}$ 2.49 Hz, H-2), 4.13 (1H, dd, $J_{5,5'}$ 12.95 Hz, $J_{4,5'}$ 1.87 Hz, H-5′), 4.41 (1H, dd, $J_{5,5'}$ 12.98 Hz, $J_{4,5}$ 1.2 Hz, H-5), 4.49 (1H, m, H-4), 4.74 (1H, m, H-3); δ_C ($CDCl_3$, 100 MHz): 24.0, 25.9 ($C(\underline{C}H_3)_2$), 34.7 (C-2), 67.7 (C-5), 71.2 (C-4), 71.5 (C-3), 109.4 ($\underline{C}(CH_3)_2$), 169.6 (C-1); *m*/*z* (APCI +ve): 173 ($M+H^+$, 100%). GC–MS: 190 ($M+NH_4^+$, 100%). Found: C, 55.78; H, 6.99; $C_8H_{12}O_4$ requires C, 55.81; H, 7.02.

4.4. 2-Deoxy-3,4-*O*-isopropylidene-L-ribose 8

Diisobutylaluminum hydride (12.9 ml, 1.0 M in toluene, 12.87 mmol) was added dropwise to the deoxylactone **7** (1.58 g, 9.19 mmol) in dichloromethane (40 ml) at −50°C. After 15 min, TLC analysis (ethyl acetate/hexane 2:1) indicated the loss of starting material (R_f 0.2) and the formation of a major product (R_f 0.3). Saturated aqueous potassium sodium tartrate (40 ml) and dichloromethane (80 ml) were added and the reaction stirred vigorously for a further 12 h. The organic layer was separated from the aqueous layer. The aqueous layer was then further extracted (dichloromethane, 2×50 ml), the organic fractions combined, dried (magnesium sulfate) and concentrated in vacuo. The resulting residue was then purified by flash chromatography (ethyl acetate/hexane 2:1) to yield the protected deoxy L-ribose **8** (1.05 g, 66%) as a clear, colourless oil. Both anomers were collected in a ratio of 3:1 and are referred to as major and minor, respectively. δ_H (200 MHz, $CDCl_3$): 1.33 (s, 3H, $C(CH_3)_2$, major), 1.35 (s, 3H, $C(CH_3)_2$, minor), 1.48 (s, 3H, $C(CH_3)_2$, major), 1.55 (s, 3H, $C(CH_3)_2$, minor), 1.76 (ddd, 1H, major H-2), 1.96–2.10 (m, 2H, minor H-2, minor H-2′), 2.22 (a-dt, 1H, major 2′), 3.57 (d, 1H, $J_{\text{major OH, major H-1}}$ 3.8 Hz, major OH), 3.63–3.74 (m, 2H, major H-5, minor H-5), 3.90–4.00 (m, 2H, major H-5′, minor H-5′), 4.09 (a-s, 1H, minor OH), 4.12–4.21 (m, 2H, minor H-4, major H-4), 4.38 (a-t, 1H, minor H-3), 4.46 (a-dt, 1H, major H-3), 5.04 (a-quint., 1H, minor H-1), 5.24 (a-quint., 1H, major H-1); δ_C (50.6 MHz, $CDCl_3$): 25.4 (major acetonide CH_3), 25.6 (minor acetonide CH_3), 27.2 (major acetonide CH_3), 28.0 (minor acetonide CH_3), 32.1 (major C-2), 32.6 (minor C-2), 60.7 (minor C-5), 62.0 (major C-5), 70.4 (major C-4), 70.7 (minor C-4) 71.2 (minor C-3), 71.6 (major C-3), 91.0 (major C-1), 91.5 (minor C-1), 108.8 (major $C(CH_3)_2$) 109.4 (minor $C(CH_3)_2$); ν_{max} (film) 3420 (OH); *m*/*z* (ESI +ve): 192 ($M+NH_4^+$, 60%). Found: C, 55.12; H, 8.12; $C_8H_{14}O_4$ requires C, 55.16; H, 8.10.

4.5. 2-Deoxy-L-ribose 2

The acetonide **8** (150 mg, 0.86 mmol) was dissolved in a mixture of trifluoroacetic acid:water (5:95/2 mL) and stirred at room temperature. After 20 min, TLC analysis (ethyl acetate) indicated formation of a new product (R_f 0.0) and no remaining starting material. This material was identical to an authentic sample of **2**, provided by CMS Chemicals.

4.6. X-Ray crystal data

A large single crystal of the lactone **5**, cut to give a fragment having dimensions approximately 0.16×0.18×0.52 mm, was mounted on a glass fibre using perfluoropolyether oil and cooled rapidly to 150 K in a stream of cold nitrogen using an Oxford Cryosystems CRYOSTREAM unit. Diffraction data were measured using an Enraf–Nonius Kappa CCD diffractometer (graphite-monochromated Mo Kα radiation, $\lambda = 0.71073$ Å). Intensity data were processed using the DENZO-SMN package.[20] Examination of the systematic absences of the intensity data showed the space group to be either $P2_1$ or $P2_1/m$. The structure was solved in the space group $P2_1$ using the direct-methods program SIR-92,[21] which located all non-hydrogen atoms. Subsequent full-matrix least-squares refinement was carried out using the CRYSTALS program suite.[22] Coordinates and anisotropic thermal parameters of all non-hydrogen atoms were refined. The hydroxyl hydrogen atom was located in a difference Fourier map and its coordinates and isotropic thermal parameter subsequently refined. Other hydrogen atoms were positioned geometrically after each cycle of refinement. A 3-term Chebychev polynomial weighting scheme was applied. Refinement converged satisfactorily to give $R = 0.0284$, $wR = 0.0378$. Crystal structure data has been deposited at the Cambridge Crystallographic Data Centre CCDC 194890.

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