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Note

A new synthesis of benzyl 2-deoxy-α-D-*arabino*-hexopyranoside

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Sanders[1] reported that an attempted synthesis of benzyl 2-deoxy-α-D-*arabino*-hexopyranoside involving the use of hydrogen chloride in benzyl alcohol was unsuccessful. By treating 2-deoxy-D-*arabino*-hexose with a boiling mixture of benzyl alcohol, benzene, and a strongly acidic, cation-exchange resin, with azeotropic removal of water, a 60% yield of a 9:1 mixture of the benzyl α- and β-glycosides was obtained[1]. The ratio of the isomers was not appreciably changed by recrystallisation.

We now report the synthesis, in 58% yield, of the pure benzyl α-D-glycoside by using hydrogen chloride in benzyl alcohol and a procedure described for the synthesis of benzyl 3-deoxy-3-fluoro-β-D-glucopyranoside[2]. The purity of the compound was verified by 240-MHz n.m.r. spectroscopy.

EXPERIMENTAL

*Benzyl 2-deoxy-α-D-*arabino*-hexopyranoside.* — Anhydrous hydrogen chloride was passed into a mixture of benzyl alcohol (50 ml) and 2-deoxy-D-*arabino*-hexose (2.5 g) cooled to —2°. When the mixture had been saturated with hydrogen chloride, it was kept at 0° for 0.5 h. Nitrogen was then passed through the solution, in order to remove most of the hydrogen chloride. The solution was then diluted with ethyl acetate (100 ml), the acid was neutralised with solid sodium hydrogen carbonate, and the solution was filtered and concentrated *in vacuo*. The residue crystallised from ethyl acetate to give the title compound (58%). After one recrystallisation from ethyl acetate, it had m.p. 129°, $[\alpha]_D^{20}$ +93° (*c* 1, water); n.m.r. data [$(CD_3)_2SO$–D_2O, 9:1; internal Me_4Si]: δ 1.3 (m, $J_{1,2a}$ 2, $J_{2a,2e}$ 13, $J_{2a,3}$ 13 Hz, H-2a), 2.0 (m, $J_{2e,3}$ 5 Hz, H-2e), 3.1 (t, $J_{4,3}$ = $J_{4,5}$ = 9 Hz, H-4), 3.6 (m, 4 H, H-3, 5, 6, 6′), 4.4 and 4.6 (2 d, 2 H, *J* 12.5 Hz, PhCH_2), 4.94 (d, $J_{1,2a}$ 2 Hz, H-1α), and 7.36 (phenyl).

Anal. Calc. for $C_{13}H_{18}O_5$: C, 61.41; H, 7.09. Found: C, 61.23; H, 7.23.

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