1-O-Formyl-β-D-glucopyranose tetra-acetate

LESLIE HOUGH AND ANTHONY W. LEWIS

Department of Chemistry, Queen Elizabeth College (University of London), Campden Hill Road, London W8 7AH (Great Britain)

(Received November 13th, 1974; accepted for publication, December 5th, 1974)

The synthesis of glycosides of methyl $18-\beta$ -glycyrrhetate by a modification of the Koenigs-Knorr synthesis, as described by Miescher and Meystre¹ for the synthesis of steroid glycosides, gave low yields. Thus, the preparation of methyl $18-\beta$ -glycyrrhet- $3-\beta$ -yl 2,3,4,6-tetra-O-acetyl- β -D-glucopyranoside, previously described by Mustafa and Fayez², gave the desired glycoside in 32% yield.

We investigated the possibility of using an alternative solvent to achieve improved yields of the acetylated glycoside. With N,N-dimethylformamide, no condensation between methyl 18- β -glycyrrhetate and 2,3,4,6-tetra-O-acetyl- α -D-glucopyranosyl bromide was observed at room temperature using mercuric cyanide as the acid acceptor. However, under these conditions, 2,3,4,6-tetra-O-acetyl- α -D-glucopyranosyl bromide was converted into 2,3,4,6-tetra-O-acetyl-1-O-formyl- β -D-glucopyranose (24% yield), m.p. 121–123°, $[\alpha]_D^{18}$ +8°; lit. 3, m.p. 121°, $[\alpha]_D^{16}$ +6°; the 1 H-n.m.r. spectrum showed a sharp singlet at τ 2.2 which could be assigned to the formyl proton. The bromide did not react with N,N-dimethylformamide in the absence of mercuric cyanide. The formic ester probably arises by a reaction scheme outlined below.

$$CH_2OAC$$
 OAC
 OAC

174 NOTE

ACKNOWLEDGMENT

We are grateful to Biorex Laboratories Ltd. for an award to A.W.L.

REFERENCES

- 1 CH. MEYSTRE AND K. MIESCHER, Helv. Chim. Acta., 27 (1944) 231, 1153.
- 2 E. A. ABU-MUSTAFA AND M. B. E. FAYEZ, Egypt. J. Chem., 2 (1959) 251.
- 3 B. HELFERICH AND R. GOOTZ, Ber., 62B (1929) 2788.