Note

Sucrochemistry
Part IV*. A direct preparation of sucrose 2,3,4,6,1',3',4'-hepta-acetate

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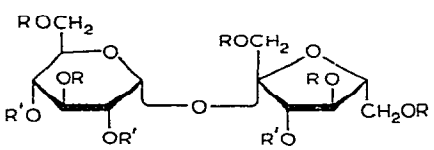
Sucrose 6'-phosphate (**1**), an intermediate in the biosynthesis of sucrose, has recently been synthesised[1] from sucrose 2,3,4,6,1',3',4'-hepta-acetate (**2**), the preparation of which has been reported in 14% and 8% yield overall from sucrose by Buchanan *et al*[1] and by Otake[2] In seeking a convenient preparation of the hepta acetate **2**, the reported[3] selective hydrolysis of steroidal primary acetates by alumina prompted us to investigate the partial de-esterification of sucrose octa-acetate (**3**) by this method When sucrose octa-acetate was adsorbed on to alumina from a chloroform solution and allowed to stand for 46 h, elution then afforded a complex mixture which was fractionated on silica gel to give the hepta-acetate **2** in 9% yield The selective removal of the primary methanesulphonyl group from monosaccharide derivatives by alumina has been described[4], and the conversion of several disaccharide octa-acetates into their hepta-acetate derivatives, by the loss of the anomeric acetyl group on treatment with piperidine in tetrahydrofuran, is also known[5]. Although the occurrence of ester hydrolysis on alumina has been known for some time[6], the use of this technique to achieve selective *O*-deacetylation of sugar acetates has been seldom reported[7]



1 R = H, R'' = PO(OH)$_2$
2 R = Ac, R = H
3 R = R'' = Ac

EXPERIMENTAL

A solution of the octa-acetate **3** (5 0 g) in ethanol-free chloroform (70 ml) was adsorbed on to a column of dry alumina (Laporte type H; 113 g) which was then allowed to stand for 46 h Elution from the alumina with chloroform–methanol (3 1) gave, on concentration of the eluate, a syrup (4 91 g) shown to be a complex

*Part III R. Khan, *Carbohyd Res*, 22 (1972) 441

mixture by t l c Chromatography on silica gel (Mallinckrodt, 350 g), with ethyl acetate–light petroleum (1 1) as eluent, afforded the octa-acetate **3** (490 mg) as the first fraction, followed by a second fraction (1 85 g) which contained at least four components The latter was further chromatographed on silica gel, using chloroform–methanol (50 1), to yield the hepta-acetate **2** (440 mg, 9%), m p 158–160° (from aqueous ethanol), $[\alpha]_D^{24}$ +52 5° (*c* 0 4, chloroform), identical (m p and mixed m p , n m r) with an authentic sample (m p 160°, $[\alpha]_D$ +49 5°) kindly provided by Professor J G Buchanan

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