tained some dissolved hydrazide besides other condensation products formed by side reactions. Further quantity of the acid was recovered by hydrolysing these products. The mother liquor was boiled under reflux with an excess of sodium hydroxide, acidified to a pH 3.5 and the precipitated acid collected by filtration. The total yield of the hydrazide, taking into consideration the recovered *iso*-nicotinic acid in both the steps amounted to over 90 % of the theory.

Procedure 2. To a solution of *iso*nicotinic acid (30 g) in 60 % hydrazine hydrate (27 ml), xylene (100 ml) was added and the mixture was heated in an oil bath at 170°. A mixture of xylene and hydrazine hydrate distilled over slowly during the first 2 h. The temperature of the oil bath was now lowered to 150° and maintained at this point for the next 4 h. During this period the last traces of xylene and a strong solution of hydrazine hydrate distilled over. The reaction product was cooled somewhat, water (20 ml) was added and the resulting solution filtered at a temperature between 70—75°. The filtrate was cooled to —3° when crude *iso*nicotinic acid hydrazide crystallised out; yield 24—25 g.

Recovery of *iso*nicotinic acid was carried out as described under procedure 1.

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A New Synthesis of 4-*O*-Methyl-D-Glucose

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The unambiguous synthetic routes leading to 4-*O*-methyl-D-glucose [1-3] are rather tedious and give low yields. As we required this substance and its methyl β-pyranoside we sought for other methods of preparing them. Bearing in mind the ease with which acyl migration takes place from the 4- to the 6-position, we investigated the direct methylation of the easily available methyl 2,3,4-tri-*O*-acetyl-β-D-glucopyranoside with methyl iodide and silver oxide. The alkalinity of the silver oxide we believed would probably be sufficient to effectively catalyse the acyl migration. This reaction has already been investigated in the α-series by Haworth, Hirst and Teece[4]. They found that methylation took place in the 2-position. On the other hand, Helferich and Lang [5] prepared methyl 2,3,6-tri-*O*-acetyl 4-*O*-methyl-β-D-glucopyranoside by the Purdie methylation of methyl 2,3,6-tri-*O*-acetyl-β-D-glucopyranoside. These results show that acyl migration is rather unpredictable and may proceed by several paths.

By methylation of methyl 2,3,4-tri-*O*-acetyl-β-D-glucopyranoside with methyl iodide and silver oxide in dimethylformamide, according to the method of Kuhn *et al.*[6], we have now obtained crystalline methyl 2,3,6-tri-*O*-acetyl-4-*O*-methyl-β-D-glucopyranoside in a yield of 45 %. A hydrolysate of the mother liquors contained 4-*O*-methyl-D-glucose as the main product, contaminated by glucose and by some methyl glucoses. 2-*O*-Methyl-D-glucose, which has almost the same electrophoretic mobility as 4-*O*-methyl-D-glucose but a higher R_F-value, was only present in traces. The pure, acetylated glucoside was converted by standard procedures to the crystalline methyl 4-*O*-methyl-β-D-glucopyranoside and to the amorphous 4-*O*-methyl-D-glucose. Paper chromatographic and paper electrophoretic examination of this latter product showed it to be indistinguishable from an authentic sample of 4-*O*-methyl-D-glucose but to be readily distinguishable from the other mono-*O*-methyl-D-glucoses. The values of the m.p. and optical rotations of the crystalline products were in good agreement with those previously recorded [1,2]. As comparatively few steps are needed, and as the yields are fairly good, this is probably the most convenient synthesis of 4-*O*-methyl-D-glucose and of its methyl-β-pyranoside.

Experimental. Methyl 2,3,6-tri-O-acetyl-4-O-methyl-β-D-glucopyranoside. Methyl 2,3,4-tri-*O*-acetyl-β-D-glucopyranoside (4.0 g) was dissolved in dimethylformamide (50 ml) and to this solution methyl iodide (12 ml) was added. Silver oxide (8.0 g) was added in four portions during 30 min under vigorous stirring and

with external cooling with tap water. The stirring was continued for 6 h, then the solution was filtered. The silver salts were washed with some dimethylformamide and, after chloroform (100 ml) had been added to the combined filtrate and washings, the solution was washed with 6 % aqueous potassium cyanide (100 ml). After washing the aqueous phase with chloroform (2 × 25 ml), the chloroform layers were combined and were washed with water (2 × 50 ml), dried over calcium chloride and concentrated. The remaining dimethylformamide was removed by co-distillation with butanol. The crystalline residue was recrystallised from ethanol, and this, together with the material obtained by working up the mother liquors, gave a total of 1.90 g having m.p. 104—105°. After further crystallisations the product had m.p. 110—111° (corr.) and $[\alpha]_D^{21}$ —35° (c = 1, in chloroform).

Methyl 4-O-methyl-β-D-glucopyranoside. By deacetylation of the above acetate the glycoside was obtained. After crystallisation from ethyl acetate it had m.p. 102—103° (corr.), $[\alpha]_D^{20}$ —18° (c = 1, in water).

4-O-Methyl-D-glucose. Acid hydrolysis of the glycoside yielded 4-*O*-methyl-D-glucose. $R_{Glucose}$ 2.40 (in butanol-ethanol-water, 10:3:5). M_G 0.17 (in 0.1 M borate buffer of pH 10). The corresponding values for 2-. 3- and 6-*O*-methyl-D-glucose were $R_{Glucose}$ 2.78, 2.67 and 2.47 and M_G, 0.17, 0.80 and 0.75, respectively.

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Preliminary Calculations of Mean Amplitudes of Vibration in *Cyclo*propane

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Reinvestigations on the structures of the *cyclo*alkanes are in progress[1]. From the radial distribution curve of *cyclo*propane the root mean square amplitude of vibration (u) has been calculated for the C—H and C—C bond distances. Least square calculations were applied to fit Gaussian shaped peaks to the peaks of the radial distribution curve. It was observed that there was a reproducible deviation from the symmetric form due to anharmonicity[2,3]. However, this effect will not be discussed here. The electron diffraction results for values of u are listed in Table 1.

Table 1. Mean amplitudes of vibration in *cyclo*propane (Å units).

Distance	El.diff.	Spectr.
C—H	0.0784 ± 0.0015	0.0770
Distance	El.diff.	Spectr.
C—C	0.0480 ± 0.0015	0.0489

It is of interest to compare these values with theoretical ones calculated from spectroscopic data. Some simple, approximate methods, which yield data of sufficient accuracy for this purpose, shall be reported in this communication.

The C—H distances. The four C—H stretching fundamental frequencies have been separated from the remaining fundamentals. Thus an explicit approximate formula for the mean square amplitude could be evaluated, the result being[4]

$$u^2_{C-H} = h\ (\mu_H + \mu_C)^{1/2} / 4\pi k^{1/2}$$

where only the principal C—H stretching force constant (k) appears. μ_H and μ_C are the inverse masses of the H and C atoms. From the experimental fundamentals[5] (A_1') 3 029, (E') 3 024.4, (A_2'') 3 103.0 and (E'') 3 080 cm^{-1}, the value