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REACTION OF MALTOSE WITH 2,2-DIMETHOXYPROPANE

YOSHIMITSU UENO, KOHSUKE HORI, RYO YAMAUCHI, MAKOTO KISO, AKIRA HASEGAWA, AND KOJI KATO
Department of Agricultural Chemistry, Gifu University, Kakamigahara, Gifu 504 (Japan)

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ABSTRACT

Acetonation at room temperature of maltose with 2,2-dimethoxypropane in *N*,*N*-dimethylformamide containing an excess of *p*-toluenesulfonic acid gave 1,2-*O*-isopropylidene-α-maltose, 4-*O*-α-D-glucopyranosyl-2,3:5,6-di-*O*-isopropylidene-D-glucose aldehydrol, 1,2:4′,6′-di-*O*-isopropylidene-α-maltose, and 1,6:3,2′:4′,6′-tri-*O*-isopropylidene-β-maltose. When this reaction was conducted at 80°, 4′,6′-*O*-isopropylidenemaltose and 3,2′:4′,6′-di-*O*-isopropylidenemaltose were formed in the presence of a trace of *p*-toluenesulfonic acid, and 1,2:4′,6′-di-*O*-isopropylidene-α-maltose and 2,3:5,6-di-*O*-isopropylidene-4-*O*-(4′,6′-*O*-isopropylidene-α-D-glucopyranosyl)-*aldehydo*-D-glucose dimethyl acetal with an excess of the acid. On the other hand, when 1,4-dioxane was used as the solvent, the 2,3:5,6:4′,6′-tri-*O*-isopropylidene dimethyl acetal derivative (**14**) was obtained in the presence of a trace of *p*-toluenesulfonic acid, and **14** and the 2,3:5,6-di-*O*-isopropylidene dimethyl acetal derivative with an excess of the acid.

INTRODUCTION

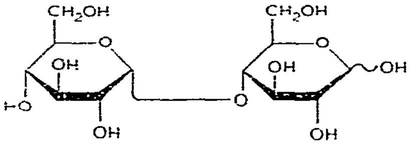
Cyclic acetal compounds have been used as intermediates in the synthesis of numerous, useful, sugar derivatives, and as intermediates for studies of conformational principles[1,2]. The acetonation of various monosaccharides[3–6] and disaccharides[7–11] with 2,2-dimethoxypropane, *N*,*N*-dimethylformamide, and *p*-toluenesulfonic acid has been studied, and it was shown that the reaction is kinetically controlled, with favored attack on the primary hydroxyl group by the reagent. We now report the results of acetonation of maltose with the 2,2-dimethoxypropane reagent under various conditions.

RESULTS AND DISCUSSION

Reaction A. — Treatment of maltose (**1**) with 6.9 mole-equivalents of 2,2-dimethoxypropane in dry *N*,*N*-dimethylformamide in the presence of *p*-toluenesulfonic acid (50 mg/g of **1**) at room temperature gave a mixture, from which four fractions (A–D) were separated by column chromatography.

Fraction A gave a single spot in t.l.c., but acetylation of the material yielded **9** and some minor products. The n.m.r. spectrum of **9** showed the presence of two acetyl and three isopropylidene groups. The coupling constant of the signals for H-1 indicated the presence of a β-bond. The mass spectrum of **9** showed the parent-ion peak at m/z 531, which was assigned to the $M^+ - CH_3$ ion. Thus, the most probable structure of **9** is 2,3′-di-*O*-acetyl-1,6:3,2′:4′,6′-tri-*O*-isopropylidene-β-maltose.

Fraction B gave a single product (**6**). The n.m.r. spectrum of the acetate (**7**) showed the presence of four acetyl and two isopropylidene groups. Its mass spectrum showed the ion peak at m/z 287, which was assigned to the di-*O*-acetyl-1,2-*O*-isopropylidene-α-D-glucose ion. Thus, the most probable structure of **7** is 3,6,2′,3′-tetra-*O*-acetyl-1,2:4′,6′-di-*O*-isopropylidene-α-maltose.



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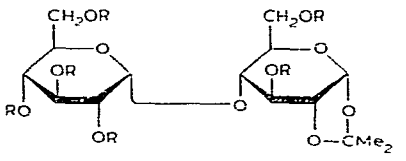

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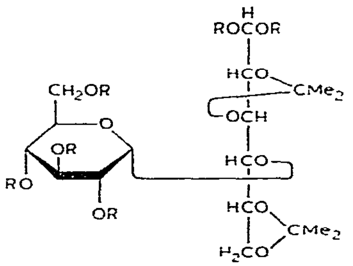

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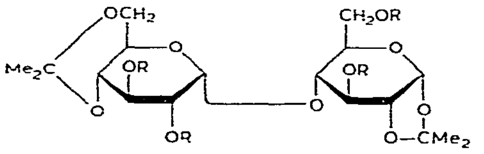

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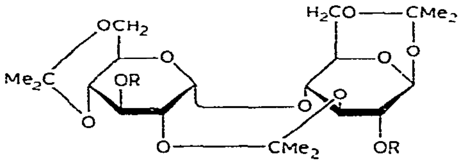


8 R = H

9 R = Ac

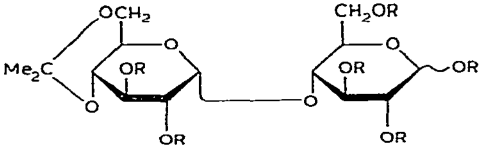
Fraction C gave a single product (**4**). The n.m.r. spectrum of the acetate (**5**) showed the presence of six acetyl and two isopropylidene groups. The chemical shift of the signal for H-1 was indicative of the presence of two acetoxyl groups on C-1. Its mass spectrum showed the ion peak at m/z 331, which was assigned to the tetra-*O*-acetyl-D-glucose ion. Thus, the most probable structure of **5** is 1,1-di-*O*-acetyl-2,3:5,6-di-*O*-isopropylidene-4-*O*-(2,3,4,6-tetra-*O*-acetyl-α-D-glucopyranosyl)-D-glucose aldehydrol.

Fraction D gave a single spot in t.l.c., and acetylation of the material yielded **3** and some minor products. The n.m.r. spectrum of **3** showed the presence of one isopropylidene and six acetyl groups. The chemical shifts and the coupling constants strongly indicated that **3** is not a 4′,6′-*O*-isopropylidenemaltose hexaacetate, but 1,2-*O*-isopropylidene-α-maltose hexaacetate.

Reaction B. — The foregoing treatment was next performed at 80° in the presence of *p*-toluenesulfonic acid (10 mg/g of **1**). Two fractions (E and F) were separated by column chromatography. Fraction E gave a single product (**12**), and acetylation of the material yielded **13** as a mixture of the anomers. From its n.m.r. and mass spectra, **13** is 1,2,6,3′-tetra-*O*-acetyl-3,2′:4′,6′-di-*O*-isopropylidenemaltose. Fraction F gave a single spot in t.l.c., and acetylation of the material yielded **11** and some minor products. The n.m.r.- and mass-spectral data for **11** indicated that it is 1,2,3,6,2′,3′-hexa-*O*-acetyl-4′,6′-*O*-isopropylidenemaltose.

Reaction C. — On the other hand, when this treatment was performed in the presence of 150 mg of *p*-toluenesulfonic acid per 1 g of **1**, three fractions (G–I) were separated by column chromatography. Fraction G gave a single product (**14**). The n.m.r. spectrum of the acetate (**15**) of **14** showed the presence of two acetyl, three isopropylidene, and two methoxyl groups, and the mass spectrum showed the ion peak at *m/z* 287 which was assigned to the di-*O*-acetyl-*O*-isopropylidene-α-glucose ion. Thus, the most probable structure of **15** is 4-*O*-(2,3-di-*O*-acetyl-4,6-*O*-isopropylidene-α-D-glucopyranosyl)-2,3:5,6-di-*O*-isopropylidene-*aldehydo*-D-glucose dimethyl acetal. Fraction H gave a single spot in t.l.c., but acetylation of the material yielded many products. Therefore, we could not obtain a single product. Fraction I was compound **6**.

Reaction D. — 1,4-Dioxane was used as the solvent, instead of *N*,*N*-dimethyl-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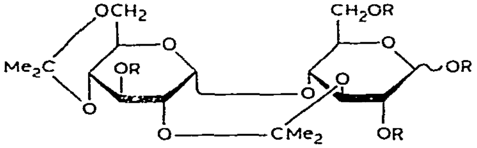
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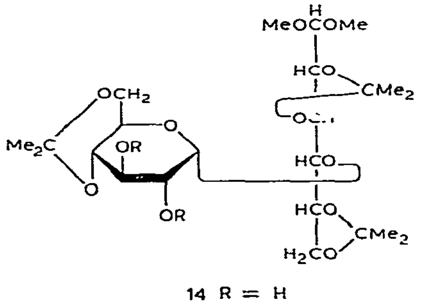
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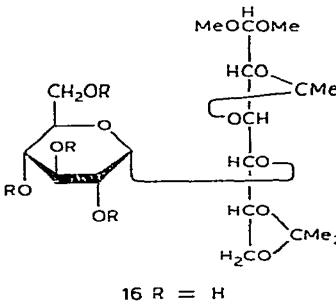
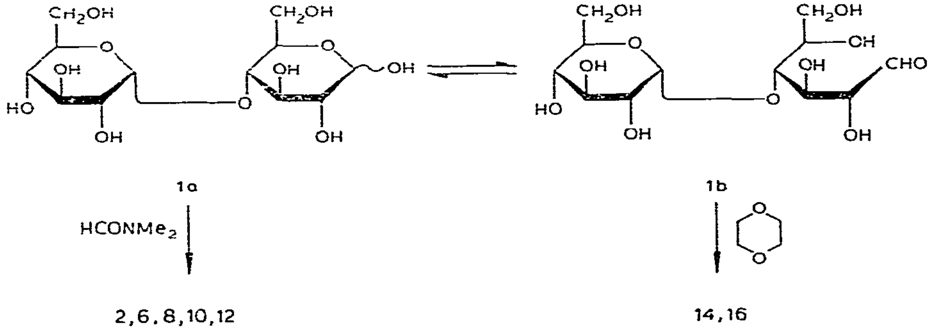

16 R = H
17 R = Ac

formamide[12]. When this treatment was performed at room temperature, the reaction did not proceed. Then, the treatment was performed at 80° in the presence of 10 mg of *p*-toluenesulfonic acid per 1 g of **1**, and one major product, **14** was obtained in 63% yield.

Reaction E. — When this treatment was performed in the presence of 150 mg of *p*-toluenesulfonic acid per 1 g of **1**, two major products, **14** and **16** were obtained, in 42 and 34% yield, respectively. The n.m.r. spectrum of the acetate (**17**) of **16** showed the presence of four acetyl, two isopropylidene, and two methoxyl groups, and its mass spectrum showed the ion peak at *m/z* 331, which was assigned to the tetra-*O*-acetyl-α-D-glucose ion. Thus, the most probable structure of **17** is 2,3:5,6-di-*O*-isopropylidene-4-*O*-(2,3,4,6-tetra-*O*-acetyl-α-D-glucopyranosyl)-*aldehydo*-D-glucose dimethyl acetal.



Scheme 1.

The behavior of maltose (**1**) with the 2,2-dimethoxypropane reagent is summarized in Scheme 1. The existence of such equilibria in the reaction solution explains the variety of products, as with D-glucose[5] and some pentoses[6]. It appears that, in *N,N*-dimethylformamide as the solvent, maltose exists almost exclusively in the pyranosylpyranose form (**1a**), and that the proportion of the acyclic form (**1b**) is small. On the other hand, products **14** and **16** were obtained when 1,4-dioxane was the solvent, indicating that maltose (**1**) exists almost entirely in the acyclic form (**1b**) in 1,4-dioxane.

EXPERIMENTAL

General methods. — Melting points were determined with a Yanagimoto micro melting-point apparatus and are uncorrected. Specific rotations were determined with a Union PM-201 polarimeter. Preparative chromatography was performed on 200-mesh, silica gel (Wako Co.) with the solvent systems specified. *N,N*-Dimethylformamide and 1,4-dioxane were distilled before use. Evaporations were conducted *in vacuo*. N.m.r. spectra were recorded at 90 MHz with a Hitachi R-22 spectrometer for solutions in chloroform-*d*; tetramethylsilane was used as the internal standard, and the sample temperature was 35°. Chemical shifts are given in δ values, and the couplings recorded are first-order spacings. Mass spectra were recorded with a Hitachi RMU-6M spectrometer operating at 70 eV.

Acetonation of maltose (**1**). — *Reaction A*. To a stirred solution of maltose (**1**) (2.0 g, 5.85 mmol) in *N*,*N*-dimethylformamide (20 mL) were added *p*-toluenesulfonic acid (100 mg) and then 2,2-dimethoxypropane (5 mL, 6.9 mol/mol of **1**). The mixture was stirred for 2 days at room temperature, and then treated with Amberlite IRA-410 (OH^-) resin (to remove the acid). The resin was filtered off, and washed with methanol, the filtrate and washings were combined, and evaporated, and the syrupy residue was chromatographed on a column (4 cm diam.) of silicic acid (50 g) with chloroform, and then four different chloroform–methanol mixtures. The 70:1 chloroform–methanol eluate yielded a syrup of **8** as a mixture (780 mg). The 50:1 chloroform–methanol eluate yielded syrupy **6** (310 mg, 12.6%). The 25:1 chloroform–methanol eluate yielded a syrup of **4** (240 mg, 9.3%), and the 10:1 chloroform–methanol eluate yielded a syrup of **2** as a mixture (630 mg).

Reaction B. To a stirred solution of maltose (**1**) (400 mg, 1.17 mmol) in *N*,*N*-dimethylformamide (4 mL) were added *p*-toluenesulfonic acid (4 mg) and then 2,2-dimethoxypropane (1.2 mL; 8.3 mol/mol of **1**). The mixture was stirred for 3 h at 80°, and then treated with Amberlite IRA-410 (OH^-) resin, to remove the acid. The resin was filtered off, and washed with methanol. The filtrate and washings were combined, and evaporated, and the syrupy residue was chromatographed on a column (2 cm diam.) of silicic acid (15 g) with two different chloroform–methanol mixtures. The 30:1 chloroform–methanol eluate yielded a syrup of **12** (170 mg, 34.4%), and the 10:1 chloroform–methanol eluate yielded a syrup of **10** as a mixture (240 mg).

Reaction C. To a stirred solution of maltose (**1**) (300 mg, 0.88 mmol) in *N*,*N*-dimethylformamide (3 mL) were added *p*-toluenesulfonic acid (45 mg) and then 2,2-dimethoxypropane (0.9 mL; 8.3 mol/mol of **1**). The mixture was stirred for 40 min at 80°, and then treated with Amberlite IRA-410 (OH^-) resin, to remove the acid. The resin was filtered off and washed with methanol. The filtrate and washings were combined, and evaporated, and the syrupy residue was chromatographed on a column (2 cm diam.) of silicic acid (10 g) with three different chloroform–methanol mixtures. The 100:1 chloroform–methanol eluate yielded a syrup of dimethyl acetal compound **14** (120 mg, 27%), and the 70:1 chloroform–methanol eluate yielded a syrupy, unknown mixture (170 mg). The 50:1 chloroform–methanol eluate yielded syrupy **6** (90 mg, 27%).

Reaction D. To a stirred solution of maltose (**1**) (300 mg, 0.88 mmol) in 1,4-dioxane (3 mL) were added *p*-toluenesulfonic acid (3 mg) and then 2,2-dimethoxypropane (0.9 ml; 8.3 mol/mol of **1**). The mixture was stirred for 15 h at 80°, and then treated with Amberlite IRA-410 (OH^-) resin, to remove the acid. The resin was filtered off and washed with methanol. The filtrate and washings were combined, and evaporated, and the syrupy residue was chromatographed on a column (2 cm diam.) of silicic acid (10 g) with chloroform and then 100:1 chloroform–methanol. The latter eluate yielded a syrup of **14** (280 mg, 63%).

Reaction E. To a stirred solution of maltose (**1**) (300 mg, 0.88 mmol) in 1,4-

dioxane (3 mL) were added *p*-toluenesulfonic acid (45 mg) and then 2,2-dimethoxypropane (0.9 mL; 8.3 mol/mol of **1**). The mixture was stirred for 4 h at 80°, and then treated with Amberlite IRA-410 (OH^-) resin, to remove the acid. The resin was filtered off and washed with methanol. The filtrate and washings were combined, and evaporated, and the syrupy residue was chromatographed on a column (2 cm diam.) of silicic acid (10 g) with two different chloroform–methanol mixtures. The 100:1 chloroform–methanol eluate yielded a syrup of **14** (190 mg, 42%), and the 20:1 chloroform–methanol eluate yielded a syrup of **16** (140 mg, 34%).

In the sections immediately following, the new products are characterized as their acetyl derivatives.

3,6,2',3',4',6'-Hexa-O-acetyl-1,2-O-isopropylidene-α-maltose (**3**). — The syrupy **2** (630 mg) was acetylated with acetic anhydride–pyridine. The product was chromatographed on a column (2 cm diam.) of silicic acid (10 g) with 200:1 chloroform–methanol, to give **3** as a syrup (550 mg, 14.8%). Compound **3** crystallized from ethanol; m.p. 166–167°, $[\alpha]_D^{20}$ +90.3° (*c* 0.6, chloroform); n.m.r. data at 90 MHz: δ 1.3 and 1.6 (2 s, Me_2C), 2.0–2.1 (6 AcO), 4.88 (d of d, $J_{1',2'}$ 4, $J_{2',3'}$ 10 Hz, H-2'), 5.05 (near t, $J_{3',4'} \simeq J_{4',5'}$ 10 Hz, H-4'), 5.07 (m, H-3), 5.45 (t, $J_{2',3'} = J_{3',4'} =$ 10 Hz, H-3'), 5.5 (d, $J_{1',2'}$ 4 Hz, H-1'), and 5.6 (d, $J_{1,2}$ 5 Hz, H-1); *m/z* 619 (4, $M^{+} -$ Me), 559 (5), 517 (5), 331 (33), 287 (36), 229 (23), 169 (50), 139 (21), 127 (35), 115 (14), 109 (60), 97 (26), 85 (24), and 43 (100, MeC^+O).

1,1-Di-O-acetyl-2,3:5,6-di-O-isopropylidene-4-O-(2,3,4,6-tetra-O-acetyl-α-D-glucopyranosyl)-D-glucose aldehydrol (**5**). — The syrup of **3** was acetylated with acetic anhydride–pyridine, and **5** was obtained as a syrup, $[\alpha]_D^{20}$ +76.8° (*c* 0.164, chloroform); n.m.r. data at 90 MHz: δ 1.35–1.5 (2 Me_2C), 2.0–2.1 (6 AcO), 4.9 (d of d, $J_{1',2'}$ 4, $J_{2',3'}$ 11 Hz, H-2'), 5.05 (m, H-4'), 5.35 (d, $J_{1',2'}$ 4 Hz, H-1'), 5.46 (d of d, $J_{2',3'}$ 11, $J_{3',4'}$ 9 Hz, H-3'), and 6.96 (d, $J_{1,2}$ 4 Hz, H-1); *m/z* 677 (10, $M^{+} -$ Me), 619 (3), 503 (3), 331 (51), 271 (11), 231 (14), 229 (15), 173 (29), 169 (80), 145 (19), 139 (21), 129 (28), 127 (47), 115 (25), 109 (60), 101 (70), and 43 (100, MeC^+O).

3,6,2',3'-Tetra-O-acetyl-1,2:4',6'-di-O-isopropylidene-α-maltose (**7**). — The syrup of **6** was acetylated with acetic anhydride–pyridine, and the product (**7**) crystallized from ethanol; m.p. 180.5–182°, $[\alpha]_D^{20}$ +82° (*c* 0.6, chloroform); n.m.r. data at 90 MHz: δ 1.35, 1.37, 1.45 and 1.6 (4 s, 2 Me_2C), 2.05–2.1 (4 AcO), 4.7 (d of d, $J_{1',2'}$ 4, $J_{2',3'}$ 10 Hz, H-2'), 5.05 (m, H-3), 5.3 (m, H-3'), 5.43 (d, $J_{1',2'}$ 4 Hz, H-1'), and 5.58 (d, $J_{1,2}$ 4 Hz, H-1); *m/z* 575 (26, $M^{+} -$ Me), 489 (4), 287 (66), 229 (44), 227 (21), 169 (80), 127 (48), 109 (66), and 43 (100, MeC^+O).

2,3-Di-O-acetyl-1,6:3,2':4',6'-tri-O-isopropylidene-β-maltose (**9**). — The syrup of **8** (400 mg) was acetylated with acetic anhydride–pyridine, and the product was chromatographed on a column (2 cm diam.) of silicic acid (10 g) with 200:1 chloroform–methanol, to give **9** as a syrup (260 mg, 15.8%), which crystallized from ethanol; m.p. 223–225°, $[\alpha]_D^{20}$ +50° (*c* 0.3, chloroform); n.m.r. data at 90 MHz: δ 1.34–1.45 (3 Me_2C), 2.08 and 2.1 (2 s, 2 AcO), 4.7 (d, $J_{1',2'}$ 4 Hz, H-1'), 5.08 (near t, $J_{1,2} \simeq J_{2,3} =$ 8 Hz, H-2), 5.39 (near t, $J_{2',3'} \simeq J_{3',4'} =$ 9 Hz, H-3'), and 6.03 (d, $J_{1,2}$ 8

Hz, H-1); *m/z* 531 (4, $M^+ - Me$), 517 (4), 515 (4), 287 (12), 229 (18), 169 (14), 127 (28), 109 (24), 101 (100), and 43 (100, MeC$^+$O).

*1,2,3,6,2',3'-Hexa-*O*-acetyl-4',6'-*O*-isopropylidenemaltose* (**11**). — The syrup of **10** (240 mg) was acetylated with acetic anhydride–pyridine, and the product was chromatographed on a column (2 cm diam.) of silicic acid (10 g) with 300:1 chloroform–methanol, to give **11** as a syrup (225 mg, 30.3%), and a mixture of several compounds (120 mg). Compound **11** crystallized from ethanol; m.p. 197–198°, $[\alpha]_D^{20}$ +14.5° (*c* 0.058, chloroform); n.m.r. data at 90 MHz: δ 1.35 and 1.44 (2 s, Me$_2$C), 2.0–2.12 (6 AcO), 4.82 (d of d, $J_{1',2'}$ 4, $J_{2',3'}$ 11 Hz, H-2'), 4.92 (d of d, $J_{1,2}$ 8, $J_{2,3}$ 10 Hz, H-2), 5.1–5.4 (m, H-3,3'), 5.28 (d, $J_{1',2'}$ 4 Hz, H-1'), and 5.72 (d, $J_{1,2}$ 8 Hz, H-1); *m/z* 619 (10, $M^+ - Me$), 331 (11), 287 (9), 229 (8), 169 (54), 127 (22), 109 (31), 101 (30), 81 (21), and 43 (100, MeC$^+$O).

*1,2,6,3'-Tetra-*O*-acetyl-3,2':4',6'-di-*O*-isopropylidenemaltose* (**13**). — The syrup of **12** was acetylated with acetic anhydride–pyridine, and the product was chromatographed on a column (2 cm diam.) of silicic acid (10 g) with 300:1 chloroform–methanol to give the anomers of **13**. The α anomer had $[\alpha]_D^{20}$ +52° (*c* 0.85, chloroform); n.m.r. data at 90 MHz: δ 1.3–1.45 (2 Me$_2$C), 2.0–2.2 (4 AcO), 4.85 (d of d, $J_{1,2}$ 4, $J_{2,3}$ 10 Hz, H-2), 5.25 (near t, $J_{2',3'} = J_{3',4'} = 9$ Hz, H-3'), 5.5 (d, $J_{1',2'}$ 4 Hz, H-1'), and 6.25 (d, $J_{1,2}$ 4 Hz, H-1). The β anomer had $[\alpha]_D^{20}$ +44° (*c* 0.01, chloroform); n.m.r. data at 90 MHz: δ 1.27–1.45 (2 Me$_2$C), 2.04–2.2 (4 AcO), 5.02 (d of d, $J_{1,2}$ 8, $J_{2,3}$ 11 Hz, H-2), 5.2 (m, H-3'), 5.45 (d, $J_{1',2'}$ 4 Hz, H-1'), and 5.65 (d, $J_{1,2}$ 8 Hz, H-1); mass-spectral data for **13**: *m/z* 575 (45, $M^+ - Me$), 517 (6), 489 (5), 359 (9), 317 (28), 287 (15), 229 (24), 184 (31), 169 (86), 142 (42), 139 (60), 127 (41), 109 (97), 101 (42), 97 (56), 69 (49), 59 (51), and 43 (100, MeC$^+$O).

*4-*O*-(2,3-Di-*O*-acetyl-4,6-*O*-isopropylidene-α-glucopyranosyl)-2,3:5,6-di-*O*-isopropylidene-*aldehydo-D-*glucose dimethyl acetal* (**15**). — The syrup of **14** was acetylated with acetic anhydride–pyridine, to yield **15** as a syrup; $[\alpha]_D^{20}$ +68° (*c* 0.114, chloroform); n.m.r. data at 90 MHz: δ 1.35–1.5 (3 Me$_2$C), 2.02 and 2.08 (2 AcO), 4.9 (d of d, $J_{1',2'}$ 4, $J_{2',3'}$ 10 Hz, H-2'), 5.15 (d, $J_{1',2'}$ 4 Hz, H-1'), and 5.37 (d of d, $J_{2',3'}$ 10, $J_{3',4'}$ 9 Hz, H-3'); *m/z* 577 (5 $M^+ - Me$), 493 (4), 303 (6), 287 (7), 227 (7), 203 (6), 169 (14), 141 (13), 127 (20), 115 (36), 101 (50), 85 (26), and 43 (100, MeC$^+$O).

*2,3:5,6-Di-*O*-isopropylidene-4-*O*-(2,3,4,6-tetra-*O*-acetyl-α-D-glucopyranosyl)-*aldehydo-D-*glucose dimethyl acetal* (**17**). — The syrupy **16** was acetylated with acetic anhydride–pyridine, and **17** was obtained as a syrup, $[\alpha]_D^{20}$ +116.7° (*c* 0.06, chloroform); n.m.r. data at 90 MHz: δ 1.35–1.46 (2 Me$_2$C), 2.00, 2.01, 2.09, and 2.11 (4 s, AcO), 4.93 (d of d, $J_{1',2'}$ 4, $J_{2',3'}$ 10 Hz, H-2'), 5.05 (t, $J_{3',4'} = J_{4',5'} = 9.5$ Hz, H-4'), 5.3 (d, $J_{1',2'}$ 4 Hz, H-1'), and 5.48 (d of d, $J_{2',3'}$ 10, $J_{3',4'}$ 9.5 Hz, H-3'); *m/z* 621 (8, $M^+ - Me$), 331 (18), 169 (65), 127 (18), 115 (40), 101 (52), 85 (20), and 43 (100, MeC$^+$O).

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